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COMPOSITES SUPPORTABILITY RAPID TEST AND EVALUATION

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Final Technical Report - October 1986-February 1990

MATERIALS LABORATORY
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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

Materials investigations were conducted in the general areas of adhesive bonding, composite materials, aircraft transparencies, and elastomeric seals. These studies were oriented towards characterization of new materials and processes, development of repair techniques, development of new processing techniques, and solution of current operational problems involving materials and processing. Some qualification testing was carried out on new materials.

Studies involved the characterization of inorganic and water-based primers for adhesive bonding, adhesive and resins for aircraft repair, novel surface preparation procedures for aluminum and composite adherends, high temperature adhesives, bonding of aluminum castings, composite paint removal techniques, techniques to prepare thermoplastic prepreg, use of induction heating to cure composites, honeycomb repair procedures, evaluation of heat lamps for curing of composite repair patches, assessment of resin transfer molding for filament wound structure, studies of battle-damage-repair techniques for aircraft transparencies, and determination of aircraft fuel and lubricating oil compatibility with elastomeric seal materials.

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Composites
Adhesives
Repair
Surface Preparation
Inorganic Primer
Water-Based Primer
High Temperature Adhesives
Paint Removal
Induction Heating
Honeycomb
Heat Lamps
Resin Transfer Molding
Filament Wound Structure
Battle Damage Repair
Aircraft Transparency
Bonding
Prepreg
Seals
Fuels
Lubricating Oil
Compatibility
Elastomers

PREFACE

This final report covers work performed during the period October 1986 through February 1990 under Air Force Contract F33615-86-C-5031, Project Number 2418. The work was administered by the Wright Research and Development Center, Materials Laboratory, Systems Support Division, Wright-Patterson Air Force Base, Ohio. Mr. Robert Urzi was the Project Engineer.

The program was performed by the University of Dayton Research Institute under the general supervision of D.A. Gerdeman, Project Sueprvisor. Personnel who made major contributions to the program include: D.R. Askins, C.W. Griffen, R.J. Kuhbander, G.W. Lawless, J.C. McKiernan, S.S. Saliba, G. Andrews, A. Behme, D. Byrge, S. Caldwell, M. Piekutowski, D. Pike, J. Stalter, and J. Wright. Jeanne Miller, Secretary, organized and typed this summary report. This report was submitted in March 1990. The contractor's report number is UDR-TR-90-24.

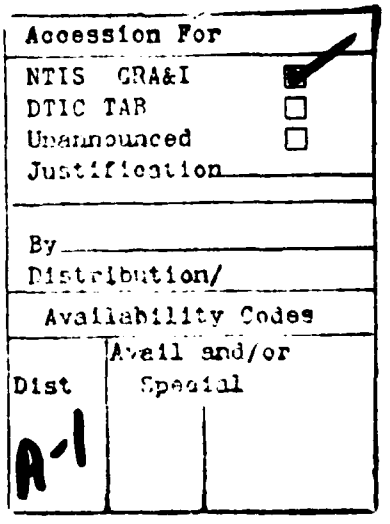


TABLE OF CONTENTS

TABLE OF CONTENTS (Concluded)

LIST OF TABLES

LIST OF TABLES (Continued)

LIST OF TABLES (Continued)

LIST OF TABLES (Continued)

LIST OF TABLES (Concluded)

LIST OF FIGURES

LIST OF FIGURES (Concluded)

1. Qualification of a Water Base Primer for F-111 Repair

A program was conducted to evaluate a water base primer, Hysol EA-9289, for use in repair of the F-111. The Sacramento Repair Depot at McClellan Air Force Base has been using Ciba Geigy's Reliabond 500 primer for adhesive bond repair of F-111 aircraft panels. The R-500 is qualified to General Dynamics Specification FMS-1013C and contains 790 G/L volatile organic compounds (VOC), which is far above the allowable limit of 340 G/L established by Sacramento County. UDRI conducted testing according to FMS-1013C, "Structural Adhesive for Honeycomb Sandwich Constuction-High Temperature Resistant," on the EA-9289 primer, which contains 35 G/L VOC.

Test Plan:

The test plan was to obtain the materials specified in the qualification specification and test to the requirements of FMS-1013C using both the existing high VOC solvent base primer and the candidate low VOC water base primer. Table 1 lists the materials, description, and sources for the materials as specified in FMS-1013C.

The tests to be conducted are given in FMS-1013C and are also listed here in Table 2. During the discussion of this project with the WRDC/MLSE Project Engineer, two concerns surfaced. The first concern was that the tests specified in FMS-1013C did not address the corrosion resistance of the primers. In general, the tests are focused on adhesives rather than primers. Because of this, two tests were added to the 14 already presented in the specification. The first was to determine the effect of salt fog on tensile lap shear strength after 30- and 90-day exposure. The second test was to determine corrosion resistance by scribing test panels with two diagonal scribe marks, extending corner to corner, and exposing them to 5% salt fog at 95°F for 30 and 60 days.

Another concern was that the water base primer, EA-9289, being evaluated did not contain corrosion inhibitors. While

corrosion inhibitors do present other environmental concerns, they have historically increased corrosion resistance. Another water base primer with a reportably low VOC, 58 G/L, was obtained for test. This primer from 3M is designated EC-3983 and was tested only to the two salt fog tests added to the program. The results were for information only.

Primer Application:

According to FMS-1013C, the primer must be applied by brush or spray coat. Most of the panels in this program were spray coated. Spray application using the solvent base primer, R-500, caused no problems. However, spraying the higher solids content water base primers was at first very difficult. The primer coating was uneven and heavy in some areas of the panel. This result is often described as "fish eyes." After some practice and direction from Hysol, an improved technique was developed and thin uniform coats were obtained. The improvements were obtained by using very high air pressure and adjusting the spray gun to a fine mist over a large area. Once the technique was mastered, uniform thin coats were easily sprayed. All primer panels, unless otherwise noted, were air dried 30 minutes at room temperature followed by 30 minutes at 250°F, as directed in FMS-1013C.

Test Panel Fabrication:

The adherend sheet material was 2024-T81 bare aluminum. This caused some problems because T81 is not a common aluminum temper. Although FMS-1013C requires an FPL (sulfuric acid/sodium dichromate) etch for the aluminum, UDRI was directed to use PAA (phosphoric acid anodization). After the panels were anodized and primed, each was assembled using the appropriate adhesive and configuration and cured according to FMS-1013C. Adhesive Forms 1 and 3 were cured 60 minutes at 350°F under 45 psi. Adhesive Form 4 was cured 3 hours at 275°F under 25 psi.

Qualification Test Nos. 1 and 3:

These tests require a laminate to be co-cured along with a layer of adhesive on the aluminum substrates. The panel is then machined into tensile lap shear specimens similar to a blister detection panel. However, according to FMS-1013C the aluminum shall be unprimed. Since the objective of this project is to compare primers, the WRDC/MLSE Project Engineer agreed not to run this test.

Qualification Test No. 2:

This test requires that tensile lap shear strength be determined for a Form 1B adhesive per Section 1.2 of FMS-1013C at -65°F, 75°F, 270°F, and 350°F. In addition, one-half of the specimens were to be conditioned for 300 hours at 270°F plus 10 hours at 350°F before testing. Tensile lap shear panels were primed with the R-500 or the EA-9289 primers, fabricated as required, and machined into tests specimens per MMM-A-132. Heat conditioning was performed on individual specimens. The results obtained using the R-500 primer are higher than those obtained using the EA-9289 primer, but all exceed the minimum requirements specified in FMS-1013C. Also, all failure modes were primarily cohesive. The results obtained are shown in Table 3.

Qualification Test No. 3:

Qualification Test No. 3 is intended to determine aluminum overlap shear creep according to MMM-A-132. Tensile lap shear panels were primed with R-500 or EA-9289 primer, fabricated, and machined as required. The adhesive used is Form 1B. The tensile lap shear coupon bond line shear stress was maintained for the loading period and at the temperature noted below:

(a) 300 hrs. at 75°F under 2000 psi;
(b) 300 hrs. at 270°F under 1700 psi; or
(c) 10 hrs. at 350°F under 1200 psi.

This task is not complete, but the results obtained to date are shown in Table 4. All of the results obtained exceed the maximum allowable creep in FMS-1013C.

Qualification Test No. 4:

This task required that honeycomb sandwich panels be fabricated and flatwise tension specimens be machined and tested at four different temperatures. Tests were to be conducted according to FPS-1028 Method B-057. This method required unique loading blocks and circular 2-inch specimens which are bonded in a recess in each block. These unique loading blocks required some design and fabrication at UDRI. At least 24 blocks would have had to be machined in order to perform the tests efficiently. These would have been very expensive. Further, since the specimens must be circular and fit into a recess in the loading block for bonding, they too would have been expensive and time consuming to prepare. Square flatwise tension tests have been successfully performed on honeycomb sandwich panels for many years. The square specimens are inexpensive to fabricate and the loading fixtures and blocks were already available. The WRDC/MLSE Project Engineer agreed to perform some flatwise tension tests using the established square specimens. If those results proved satisfactory, then preparation and testing would proceed using the square specimen design.

A honeycomb sandwich panel for flatwise tension tests was fabricated using the Form 1B adhesive, R.B. 398 N.A., and R-500 primer. One-inch square specimens were machined and bonded to loading blocks using HT-424 adhesive film. Flatwise tension tests were conducted at 75°F and 350°F. The minimum requirement for a 2-inch-diameter specimen is 3475 lbs. at 75°F and 2100 lbs. at 350°F. The failure loads obtained for the 1-inch square specimens were adjusted for the same area as a 2-inch-diameter specimen and are 7417 lbs. at 75°F and 4314 lbs. at 350°F. The results far exceed the minimum requirements presented in FMS-1013C. Based upon these satisfactory results, the WRDC/MLSE Project Engineer gave permission to proceed with the square specimens.

Honeycomb sandwich panels were then fabricated with aluminum skins primed with the R-500 or EA-9289. Individual 1-inch square specimens were then machined from the panels. One-half of the specimens were heat aged as required for 300 hrs. at 270°F plus 10 hrs. at 350°F. HT-424 adhesive was used to bond loading blocks onto individual specimens, and the cure used did not exceed the cure temperature of the adhesive used to fabricate the sandwich panels. The results obtained are shown in Table 5. All of the results exceeded the minimum requirement in FMS-1013C.

Qualification Test No. 5:

This qualification test is to determine the "Short Beam Sandwich Shear" strength in accordance with test method B-053 in specification FPS-1028. The sandwich panels were constructed according to Para. 9.3 in FMS-1013C. The aluminum honeycomb core was the same as that used for flatwise tension in qualification test No. 4, Hexcel 1/8 5052/8.1. The adhesive is Type 1B and both R-500 and EA-9289 were used to prime the skins. Short beam shear tests were conducted at -65°F, 75°F, 270°F, 350°F, and 400°F. In addition to meeting the minimum requirement in the qualification specification, all failure modes must be in core shear. Adhesive delamination is not acceptable. All of the results obtained are shown in Table 6 and meet the minimum requirements in FMS-1013C, including failure modes.

Qualification Test No. 6:

Qualification Test No. 6 is to determine "Long Beam Sandwich Shear Strength" in accordance with test method B-060 in FPS-1028 using adhesive Type 1B, R.B. 398 N.A. The sandwich panels were constructed according to Para. 9.3 in FMS-1013C using the same aluminum honeycomb core as in the two preceding tests. Long beam shear tests were conducted at -65°F, 75°F, 270°F, 350°F, and 400°F. In addition to meeting the minimum load requirement in the specification, all failure modes must be in core shear. The results obtained are shown in Table 7, and all the specimens met the minimum requirements in FMS-1013C, including failure modes.

Note that at -65°F, 75°F, and 400°F the average value reported for the EA-9289 primer is for two specimens while all others are an average of three as required in the specification. During specimen machining, three specimens were unintentionally machined in the wrong direction. Since these specimens are constructed of honeycomb, the shear results obtained are dependent upon the direction of the core, which for these tests is to be in the ribbon direction. These specimens were not replaced because all of those properly tested exceeded the minimum requirement and there was little test data scatter.

Qualification Test No. 7:

Qualification Test No. 7 is to determine "Sandwich Beam Creep" at 270°F and 300°F. Honeycomb sandwich panels were constructed according to Para. 9.3 in FMS-1013C using Hexcel aluminum core, 1/8 5052/8.1. Sandwich panel skins were primed with R-500 or EA-9289 primer as required and adhesively bonded with Type 1B adhesive, R.B. 398 N.A. Testing was in accordance with test method B-061 in FPS-1028. Special test fixturing was designed and fabricated just for this creep test. Also, a specially designed oven was purchased which would fit in the small available space between the Arcweld creep frame cross-head and frame yet be wide enough to contain the sandwich beam specimens. All of the results obtained were within the requirements in FMS-1013C and are shown in Table 8.

Qualification Test No. 8:

According to Qualification Test No. 8 per FMS-1013C, the adhesive weight loss shall be determined for adhesive Forms 1B and 3. At the request of the WRDC/MLSE Project Engineer, the weight loss was determined even though this test does not involve primers. The tests were completed per FPS 1028 Method B-005 and the results are shown in Table 9. The weight loss measured is far less than the maximum allowable per FMS-1013C.

Qualification Test No. 9:

This Qualification Test requires honeycomb sandwich panels be fabricated and subjected to a fluid tightness test. FMS-1013C requires that only Form 1B adhesive be used. Both the R-500 and XEA-9289 primers were tested. The panels were fabricated with the Hexcel aluminum honeycomb core and machined into 3-inch by 3-inch specimens. There was no sealant of any type applied to the edge of the specimens. The fluid tightness test was then conducted to FPS-1028, Method B-059 as follows:

> Completely immerse specimens in dyed JP-4 fuel (specification MIL-J-5624) maintained at 75°F±5°F for 48 hours, and specimens in dyed JP-4 fuel maintained at 180 ± 5°F for 48 hours. Both JP-4 fuel containers were pressurized to 30 PSI throughout the duration of the test. No sealant of any kind shall be applied to the edges of the specimens prior to immersion. At the end of the required immersion time, examine each specimen for evidence of fuel penetration. The specimens were visually examined for fuel penetration by cutting through the center of the core. Specification FMS-1013C requires that fuel penetration not exceed 0.50 inch.

The dye added to the JP-4 fuel is visible under a black light. Once the test was complete, the specimens were cut in half and observed under a black light for fuel penetration. There was no fuel penetration observed in any of the specimens using the solvent base primer, R-500. Also, there was no penetration observed in specimens using the XEA-9289 water base primer at 180°F. However, two of the specimens had penetration at room temperature. One of the specimens had penetration exceeding the maximum requriement in FMS-1013C. The specification is not clear regarding the requirements as to whether the maximum penetration is for individual specimens or for the average of the three specimens. If the average is used, the room temperature tests also passed. Since it did not appear logical that the test would fail at room temperature and pass at 180°F, and since at room temperature one of the specimens had no penetration, it was decided after some discussion with the WRDC/MLSE Project Engineer to repeat the room temperature test but increase the number of specimens to six.

There was no fuel penetration observed in any of the specimens upon repeat. The original and repeat results are shown in Table 10.

Qualification Test No. 10:

Qualification Test No. 10 is to determine aluminum overlap tensile shear strength at 75°F, 270°F, and 350°F. The tests were conducted according to MMM-A-132 as required in FMS-1013C. While FMS-1013C calls for the test to be conducted using Form 3 or 4 adhesive, the WRDC/MLSE Project Engineer suggested the test be conducted for both adhesive forms. Both Forms 3 and 4 are the same adhesive, AF-130/2, but the cure cycle is different. Specimens were consequently fabricated with aluminum primed with R-500 or XEA-9289 and bonded with Forms 3 and 4 adhesive. The results, shown in Table 11, all exceed the minimum requirement in FMS-1013C.

Qualification Test No. 11:

Qualification Test No. 11 consists of performing honeycomb sandwich flatwise tension tests on Form 3 adhesive. Nonmetallic core conforming to FMS-1022, Class 1, Type C was specified in FMS-1013C. The honeycomb core used was from Hexcel, HRP-3/16-7. While honeycomb sandwich panels fabricated with a Form 1B adhesive required only one layer for each skin, two layers of adhesive are required for each skin when using Form 3 adhesive (per FMS-1013C, Para. 9.2.6). As in qualification Test No. 4, the tests were to be conducted according to FPS-1028 Method B-057. This method required the 2-inch-diameter specimens and the unique loading blocks. As in test No. 4, the WRDC/MLSE Project Engineer agreed to use the 1-inch square specimens, but close attention was given the failure mode to insure that the results would be valid. Honeycomb sandwich panels were fabricated, 1-inch square specimens were machined and tested. The results were then extrapolated to the cross-sectional area of 2-inch-diameter specimens. One-half of the specimens required temperature conditioning before testing of 300 hrs. at 270°F plus 10 hrs. at 350°F. The results obtained

are shown in Table 12 and all exceed the minimum requirement in FMS-1013C.

Qualification Test No. 12:

Qualification Test No. 12 requires that short beam sandwich shear strength be determined using Form 3 adhesive, AF-130/2, and nonmetallic core, HRP-3/16-7. Honeycomb sandwich panels were fabricated using R-500 and XEA-9289 primers and two layers of adhesive as required in FMS-1013C. The tests were conducted according to FPS-1028, Method B-053 at -65°F, 75°F, 270°F, and 350°F. One-half of the specimens were heat aged for 300 hrs. at 270°F plus 10 hrs. at 350°F before testing. Another requirement is that all failure modes be in core shear; no delamination is acceptable. All of the results obtained met the requirements in FMS-1013C and are shown in Table 13.

Qualification Test No. 13:

Qualification Test No. 13 requires a laminate be co-cured with a layer of adhesive on the aluminum substrate. The panel was then to be machined into tensile lap shear specimens similar to a blister detection panel. However, according to FMS-1013C the aluminum shall be unprimed. As with Test No. 1, the WRDC/MLSE Project Engineer agreed not to run this test.

Qualification Test No. 14:

Qualification Test No. 14 requires that short beam sandwich shear strength be determined using Form 4 adhesive, AF-130/2, and nonmetallic core, HRP-3/16-7. Honeycomb sandwich panels were fabricated with skins primed with R-500 and XEA-9289. The panels were fabricated according to FMS-1013C; however, the number of adhesive layers to be used for Form 4 is not clear. According to Paragraph 9.2.6, the procedure is to "apply one layer Form 1B or two layers Forms 3 of adhesive to each primed sheet material." Since Forms 3 and 4 are actually the same adhesive but use different cure cycles, UDRI assumed that two layers should be used

for Form 4. Further justification is that the film weight for Forms 3 and 4 is low and the additional material would help form good fillets when fabricating honeycomb sandwich panels. The short beam sandwich shear strengths were determined according to FPS-1028, Method B-053 and are shown in Table 14. All of the results exceed the minimum requirements in FMS-1013C.

Test No. 15:

Test No. 15 is not a qualification test but one which was added by the WRDC/MLSE Project Engineer. The qualification specification, FMS-1013C, does not address the corrosion resistance of primers. The tests required in the specification are more directed toward the compatibility between primer and adhesive. The effect of salt fog aging per ASTM B-117 was determined on tensile lap shear strength and upon scribed aluminum panels after 30- and 90-day exposures. For this salt fog aging, three primers were evaluated: R-500 solvent base primer with 790 G/L VOC, XEA-9289 water base primer with 35 G/L VOC, and EC-3983 water base primer with 170 G/L VOC. The two water base primers differ in the percent solids, XEA-9289 has 30% and EC-3983 has 20%, and in the manufacturers' recommended cure temperature, XEA-9289 is 350°F and EC-3983 is 250°F. The SM-ALC has asked that the cure temperature in this study be limited to 250°F, which is used for the current solvent base primer, R-500.

Aluminum overlap tensile shear specimens were prepared according to MMM-A-132 using the three candidate adhesive primers and the three adhesive forms per FMS-1013C. Unaged specimens were tested for control purposes. Tests were also conducted after aging for 30 and 90 days at 95°F in a 5% salt fog according to ASTM B117. The results obtained are shown in Table 15. The data obtained is somewhat scattered, but it appears that the salt fog aging had little detrimental effect upon lap shear properties. In fact, in the case of the Forms 3 and 4 adhesive, the salt fog exposure was actually quite beneficial. This result is probably the result of some additional postcuring effects.

Primed aluminum panels were prepared to determine the effect upon unbonded surfaces after 30- and 90-day salt fog exposure. The primed panels before exposure are shown in Figures 1-3. Note that intersecting diagonal lines are scribed from one corner to another. All panels were primed on both sides. The R-500 panel was spray coated on both sides, but the water base primers were spray coated on one side and brush coated on the other side. Note that with the water base primers, especially the XEA-9289, the fish eye effect is much more apparent with the spray application. As noted earlier, this effect was eliminated once the proper spray technique was developed after consulting with Hysol.

The scribed aluminum panels were subjected to salt fog exposure for 30 and 90 days. The panels after 30-day exposure are shown in Figures 4-6. The panels primed with R-500 and EC-3983 show little corrosion and no peeling, blistering, or cracking. However, the panel primed with XEA-9289 shows a considerable amount of corrosion and loss of primer. It appears the primer is washing away during the salt fog exposure. Figures 7-9 show the panels after 90 days exposure to salt fog. There is some slight corrosion visible on the R-500 primed panel but none on the EC-3983 panel. Once again the XEA-9289 primed panel had a lot of corrosion and loss of primer.

After salt fog aging the panels primed with XEA-9289 showed considerable corrosion and loss of primer. These, however, were cured at 250°F, even though the manufacturers' recommended cure is 350°F. As a result, additional panels were primed with XEA-9289 and cured using the 350°F cure recommended by the manufacturer. These were scribed and exposed to salt fog for 30 and 90 days. After both the 30-day and 90-day exposure, no loss of primer was observed. Further, only slight corrosion was evident and no blistering or cracking was observed. The exposed panels are shown in Figures 10 and 11.

Summary:

The low VOC water base primer tested, Hysol's XEA-9289, does meet all the qualification tests per FMS-1013C. However, based upon the additional salt fog tests added, if primed aluminum is exposed to a corrosive environment and cured at 250°F, it may be susceptible to corrosion and loss of primer. UDRI contacted the primer manufacturer, Hysol, and they agreed a problem could exist if the cure temperature is 250°F. Hysol indicated that a cure temperature less than 350°F may be suitable, but it has not been established. The EC-3983 water base primer from 3M did show good resistance to salt fog exposure when cured at 250°F.

We feel that FMS-1013C does not adequately address the corrosion resistance of primers, but only the compatibility with adhesives. Additional tests for salt fog exposure, humidity, and durability would be beneficial.

2. B1-B Repair Adhesive

A program to generate design allowable data on an adhesive for repair on the B1-B was conducted. A two-part paste adhesive from Hysol designated EA9394 was selected. For repair applications, two-part paste adhesives offer several advantages. Among these are the ability to cure at room temperature or at moderately elevated temperature, low pressure cure which eliminates the need for autoclaves, fill void areas due to nonsimilarity in shape, and, due to low temperature cures, the ability to bond materials with dissimilar coefficients of thermal expansion. EA9394 is a two-part paste adhesive which contains no asbestos or MDA, has high temperature performance when cured at room temperature to 200°F, has good pot life and long-term storage at room temperature and moderate elevated temperature, and is low in toxicity.

Materials and Processing:

Numerous test variables were included in this program and are listed in Table 16. Most of the variables are evaluated by means of metal-to-metal tensile lap shear or floating roller peel

specimens. The metal used for adherends was 2024-T3 aluminum. The surface preparation for the aluminum was phosphoric acid anodization (PAA). The prepared aluminum surfaces were primed with BR-127 corrosion inhibiting primer from American Cyanamid. Most of the test panels required bondline thickness control and this was accomplished by the addition of scrim cloth, No. 2006 Subcode 701 Reemay spunbonded polyester. Generally, bondline thickness was maintained between 5-7 mils. Tensile lap shear strength was tested in accordance with ASTM D1002 and MMM-A-132, and floating roller peel in accordance with ASTM D3167. The cure cycle used full vacuum. This cure cycle was chosen based upon discussion with Hysol, previous work published, and discussion between UDRI and the WRDC/MLSE Project Engineer.

During the program, numerous unexpected developments delayed progress. Work was performed in 5 of the 15 task areas proposed. Most of the unexpected developments were encountered during Task 1, Minimum Cure, and Task 2, Control.

Task 1 - Minimum Cure:

Originally, the minimum cure study was intended to determine the time needed to reach 100%, 90%, and 80% cure for specific temperatures by means of isothermal DSC at those temperatures. Panels would then be fabricated at those temperatures for the cure times determined by DSC. However, as data became available, it was obvious that the DSC curves would be greatly affected by the heat-up rate and the starting temperature. Bonded tensile lap shear panels were fabricated according to the procedure generally used throughout the screening program. The heat-up rate achieved when bonding panels was monitored and determined to be near 1°C/min. Isothermal DSC tests were then conducted at 52°C (125°F), 66°C (150°F), 79°C (175°F), and 93°C (200°F) with a heat-up rate at 1°C/min. The time to reach 100%, 90%, and 80% of full cure was then determined at each temperature. The tabulated results are shown in Table 17. Upon examination of the time at temperature to reach a specific degree of cure, two observations were made. First, it would be extremely difficult to fabricate

bonded panels at 80% and 90% of cure at a specific temperature because the time for each is nearly the same. Also in some cases the sample reached the designated percent cure before the isothermal temperature was reached.

Second, the time at temperature for a specific percent of cure and the heat of reaction were not in a progressive order for the four test temperatures. The heat of reaction obtained at 150°F and 175°F were nearly the same and, therefore, it appears that just a slight change in the DSC baseline may result in a change in heat of reaction significant enough to change its position in line.

A fresh sample was tested at 150°F and this time the heat of reaction was nearly equivalent to that obtained at 125°F (Table 17). We do not know why the heat of reactions do not progress from low to high in the same order as the isothermal temperatures, nor why two supposedly identical tests can produce such different results. Perhaps the high percent of aluminum filler in the adhesive has some effect.

Another approach was used to determine the percent of cure at a specific temperature. Rather than determining the 80%, 90%, and 100% cure for the adhesive at a specific temperature, the percent of total cure that can be achieved at a specific temperature was determined and the results are shown in Table 18. First, the isothermal DSC runs were completed at a specific temperature, then the same sample was subjected to a dynamic DSC so that any residual cure could be determined. The total exotherm or heat of reaction was then determined and is shown in Table 18. However, here too some lack of order is observed and may again be caused by the high percent of filler. It was then obvious that another approach should be taken.

After some thought, it seemed that the minimum cure could best be expressed by determining and reporting what cure time would be required to obtain full adhesive strength for a particular cure temperature. The "standard cure" in this project is 1 hr. @ 200°F. It was determined how long it is necessary to cure

panels at R.T., 125°F, 150°F, and 175°F to obtain the same adhesive strength as with the "standard cure." Tensile lap shear panels were fabricated and tested for cure times of 1 and 2 hours at 125°F, 150°F, and 175°F, and for 24, 72, and 168 hrs. at room temperature. The results are shown in Table 19. After 1 hr. at temperature, none of the cure temperatures yielded strengths near that for the standard cure. However, after 2 hours at both 150°F and 175°F, the tensile lap shear results obtained are equal to or even slightly better than that obtained for the standard cure. Therefore, minimum cure has been established for those cure temperatures. Additional cure is apparently required for 125°F and should be further investigated. Results have also been obtained for cures at R.T. but are not as expected. The lap shear strength obtained after a 24-hour R.T. cure appear reasonable, but those after 72 and 168 hours are much lower and these should be rerun. Floating roller peel data were obtained for those temperatures where full cure has been established which are 200°F, 175°F, and 150°F. These results are also shown in Table 19. Interestingly, the peel *strengths* obtained for cure temperatures of 150°F and 175°F are significantly higher than after a 200°F cure.

Task 2 - Controls:

The standard cure cycle chosen for the control data was 1-hour at 200°F (93°C) under full vacuum. This was chosen based on recommendations by Hysol, previous work published, and discussion with the WRDC/MLSE Project Engineer. The tensile lap shear bondline thickness control method chosen was scrim cloth.

The initial lap shear specimens that were fabricated gave an R.T. ultimate strength of 3134 psi. Examination of data from Hysol and some previous work at UDRI indicated that a strength of about 4000 psi should be achievable. Further examination indicated that the cure cycles may not have been the same as currently being used. Further, the reported data were for bonds in which glass beads were used for bondline thickness control. Additional panels were fabricated using both beads and scrim for bondline

control and varying cure cycles. The major changes in the cure are the method for and amount of pressure, and cure temperature. The results obtained are shown in Table 20.

Once these data had been obtained, it appeared that the amount of pressure applied had a significant effect upon the lap shear strengths. It seemed then that optimum repairs could be obtained by simply controlling the vacuum pressure level. However, the WRDC/MLSE Project Engineer pointed out that this would be difficult for depot repair and perhaps uncontrollable for field repair. Upon further discussion, we determined that only full vacuum should be considered and that some other controllable parameter should be changed. It was suspected that if either the vacuum pressure and/or the temperature were applied too soon, the amount of adhesive in the bondline or the amount of porosity would be greatly affected. Regardless of the exact cause and effect, we feel that a time delay before the temperature and pressure are applied may have a similar effect as a reduced pressure.

Additional adherends were prepared and the adhesive applied in the usual manner except that there was a delay in the time at which the temperature and/or pressure was applied. The lap shear results for these panels are shown in Table 21. As the data indicate, a slight delay in time for applying both pressure and temperature results in a significant increase.

Also, since the reduced vacuum pressure did affect the lap shear rseults, some additional bonded panels were fabricated with varying amounts of vacuum pressure. These results are shown in Table 22. It does appear that a reduction in pressure improves the tensile lap shear properties. All of the panels were fabricated without a delay in application of pressure or temperature.

As a result of the testing described above, fabrication and testing of the tensile lap shear control specimen was begun for all three batches of adhesive. The cure cycle chosen was 1-hour at 200°F (93°C) under full vacuum pressure with a 45-minute delay of both pressure and temperature application.

All of the control tensile lap shear tests were completed and are shown in Table 23. The results at both R.T. and 200°F (93°C) are very good for all three batches of adhesive. The failure modes of the R.T. tests are primarily between the adhesive and primer. This has been true for all R.T. tests regardless of the fabrication method used. The failure mode for all 200°F tests is 100% cohesive.

All of the control floating roller peel specimens were also fabricated and tested, and the results are also shown in Table 23. The same fabrication technique was used in fabricating the peel panels as with tensile lap shear panels. This included the use of BR-127 aluminum primer and scrim cloth for bondline control. Examination of the peel results indicate that the data does not agree with that reported by Hysol, whose results are nearly double that obtained at UDRI. The results obtained here do agree with those previously reported by UDRI in some preliminary work. This was discussed with Hysol and three differences were noted: (1) Hysol's "standard cure" is 168 hours at R.T., (2) Hysol does not use scrim cloth, and (3) Hysol does not use BR-127 primer.

At this point, all was apparently going well and considerable progress has been made when a series of unexpected difficulties took place. First, the laboratory work changed hands owing to a change in personnel at UDRI. This is significant because we felt that some of the difficulties were related to processing techniques, which later proved to be false.

Work was being conducted on several concurrent tasks, including that for minimum cure. This is significant because the floating roller peel data obtained for cures at 150°F and 175°F were far better than those being obtained for the "standard cure," 1 hour at 200°F. This cast some doubt on the cure cycle selection. After obtaining these results, Hysol was contacted. We concluded that the higher cure temperature was causing the adhesive to become more brittle, which results in reduced peel strength.

Most of the panels fabricated to this point were vacuum bagged in a Zip-Vac container and cured in an oven under vacuum pressure. The vacuum pressure was being monitored by a gage between the pump and the vacuum bag. This, as well as some of the existing plumbing, did not seem to be good practice. Considerable changes were then made in the vacuum system. Additional panels were then fabricated using the "standard cure." It should also be noted that the adhesive being used by this time was about 10 months old. The results being obtained at this time were not nearly as high as those that had been obtained with the old vacuum system, original personnel, and fresher adhesive. Several panels were fabricated and each had poor adhesive lap shear strength as indicated in Table 24. The results and failed specimens were closely examined, and two observations were made. The bondline thicknesses were thicker (0.012) than normal (0.005-0.007) and the adhesive bondline was foamy. Close instructions were given the new lab personnel, and the results were improved but still unsatisfactory. The tensile lap shear results were slightly higher but still not what were once obtained. The bondline thickness was near what was expected, 0.007 inch, but the bondline remained foamy. After additional thought it was believed that the problem was then related to the improvements in the vacuum system and what was believed to be full vacuum during the original work probably was not. An additional panel was then fabricated with reduced vacuum and the revised vacuum system. The tensile lap shear results were still not as good as once obtained, but the bondline was very dense. It was then concluded that the improvements in the vacuum system were indeed causing foamy bondlines but that this was only partly responsible for the lower tensile lap shear strengths. Also contributing to lower strengths may be the age of the adhesive.

A small quantity of fresh EA9394 adhesive was obtained from MLSE and bonded panels were prepared with varying cure cycles. The tensile lap shear results obtained were compared to previous data and are shown in Table 25. It may not be possible to duplicate all of the original data because it is not known for certain

what vacuum pressure was actually in the vacuum bag. However, using fresh adhesive and reduced vacuum does produce dense bondlines and tensile lap shear near what was originally obtained. Further, if the cure temperature is also reduced, excellent results were obtained. These data are also shown in Table 25.

At this time, we concluded that full vacuum does produce foamy bondlines and reduced tensile lap shear data, although by a delay in processing, at least some of this can be overcome. The WRDC/MLSE Project Engineer suggested that bonded panels be fabricated but be isolated from the vacuum. An experiment was devised to verify that exposure of the bondline to full vacuum would result in foamy bondlines while isolating the bondline from full vacuum would produce good dense bonds. Using a fresh lot of adhesive, panels were fabricated with dead weight load (no vacuum), full vacuum, and full vacuum but in an envelope bag. An envelope bag is one which contains the entire bonded panel and is sealed so that the bondline can be compacted by the vacuum pressure but isolated from the vacuum. Further, the panels were fabricated using three bondline pressures: 5, 10, and 15 psi. Tensile lap shear specimens were machined and tested at room temperature and 200°F. The results shown in Table 26, verify that exposure of the bondline to full vacuum will result in reduced shear strengths. All of the panels fabricated with dead weight load and an envelope bag have excellent shear strengths at R.T. and 200°F. Panels fabricated using the conventional bagging technique, which exposes the bondline to vacuum, have poor shear strengths at both 10 and 15 psi. Under reduced vacuum of 5 psi, the strengths are similar to those achieved with dead weight loading or envelope bagging.

Based upon the results of panels fabricated with dead weight load, full vacuum, and full vacuum plus envelope bag, at least three options should be reviewed by MLSE before additional work proceeds: (1) discontinue the work to generate design allowable data, (2) obtain three new batches of adhesive from Hysol, quickly determine an optimum cure which would include an envelope bag and

proceed with the original work plan, and (3) perform a new adhesive screening test program and make sure the best possible adhesive candidate is being evaluated for B1-B repair.

During the same time period that the minimum cure and control data was being generated, work was proceeding on additional tasks and is discussed in the following subsections.

Task 3 - Humidity Exposure:

All of the humidity exposure tensile lap shear and floating roller peel specimens have been fabricated, aged, and tested. The humidity exposure was for 30 days at 140°F and 95-100% R.H. The tensile lap shear results are shown in Table 27 and the floating roller peel in Table 28. There is a slight reduction in tensile lap shear strength at R.T. and 200°F and a slight increase in strength at -65°F. The floating roller peel strengths are nearly the same as those obtained dry at -65°F and R.T., and a slight decrease is noted at 200°F. All of these specimens, both tensile lap shear and peel, were prepared with BR-127 primer and scrim cloth. The cure cycle was 1 hour at 200°F, with what was identified as full vacuum but probably less, and a 45-minute delay before temperature and pressure was applied.

Task 12 - Pot Life:

This task is intended to be a guide to determine what time period can elapse once the adhesive is mixed and still obtain good lap shear and peel properties. To date, tensile lap shear panels have been fabricated with 4-, 8.5-, and 18-hour hold times after adhesive mixing and application, but before assembly and cure. After a 4-hour open assembly hold time period, the tensile lap shear properties are not as high as those obtained using the "standard cure," but are considered satisfactory. Other shorter open assembly times are recommended.

Task 11 - Storage and Elevated Temperature Aging:

The purpose of this task was to determine how the reactivity of the material changes with time for a range of storage temperatures. Samples of Parts A and Part B were stored at room

temperature, 100°F, and 120°F. Both physical properties and mechanical properties were determined initially and after 1, 6, 9, and 12 months.

The physical properties did not show much change due to aging at any temperature; however, they are difficult to interpret. There does seem to be a slight increase in viscosity for both Parts A and B. Also after about 9 months at 120°F, Part B did omit a strange odor.

Tensile lap shear was determined at room temperature and 200°F. This task started as soon as the adhesive arrived at UDRI. Therefore, the initial lap shear panels were fabricated before the effects of a delay during cure or the effects of full vacuum were known. Also, those panels fabricated up to the 6-month storage period may have had less than full vacuum but greater than that which reduces shear strength. The panels fabricated after 9 months storage were processed with full vacuum. Most of the lap shear strengths measured were low, as would be expected since they were generated before all the effects of various processing parameters were known. These results are shown in Table 29. At the 12-month aging date, the effects of vacuum had become known. Panels were fabricated using the same procedure as for the previous storage periods. The tensile lap shear strengths were low, as one would predict. However, panels fabricated using full vacuum but an envelope bag had excellent tensile lap shear strengths at room temperature and 200°F using adhesive stored at all three temperatures as shown in Table 30. These data indicate that EA9394 adhesive has excellent storage life at temperatures up to 120°F. It is recommended that the task include at least 18-month storage and perhaps 24 months.

3. Surface Preparation Studies for Adhesive Bonding

A variety of approaches were pursued to develop improved surface preparations for the adhesive bonding of aluminum and composite adherends in an aircraft repair environment. The wedge test was used frequently as the criterion of performance.

The use of silane coupling agents (Dow Chemical) was explored with modest success. There are many variables involved including time, purity and concentrations of solutions, shelf life, and temperature. While Dow reported good results with these coupling materials, their data was based upon experiments with glass microscope slides. All of our work was carried out on aluminum adherends. Results obtained on aluminum were mixed. It was finally concluded that this approach was not feasible for field repair because the many variables that influence the results would be impossible to control in a field environment.

The Windecker surface preparation method was also investigated, one of its primary advantages being that it is non-acidic. This technique consists of a wet-mechanical abrasion of the surface to be bonded while it is covered with an adhesion promoting solution. The purpose of the solution is to prevent contact of the freshly abraded aluminum surface with air so that the bare aluminum surface will not be acidized. Numerous sets of aluminum wedge specimens were prepared using this technique. Phosphoric acid anodization was used as a control surface preparation method for comparison purposes. Some typical data from these tests are presented in Table 31. Additional data were generated that incorporated many controlled variables in the process (varying times of immersion, different solvents/solutions, abrasion studies, use and absence of primers and anodization). The most general conclusion reached is that phosphoric acid anodization with BR-127 primer remains the best aluminum surface preparation method. The best combination of parameters found with the Windecker procedure produced durability (wedge-crack-propagation) results that approached but did not equal those achievable with the PAA/BR127 process. Much of the adhesive data generated in this project proved useful in establishing test parameters for other efforts.

4. Alodine Surface Preparation

Alodine is an immersion surface treatment, primarily for aluminum, that is used as a paint base. The objective of this

project was to evaluate the alodine process for adhesion bonding. The supplier of the material is Amchem. "Alodine" is really the tradename, and the process involves a chromium conversion coating with hexavalent chromium and cyanides as waste by-products. The immersion solution can be acidic or basic, and there is also a nonchromium version. Wedge test results were relied on for the primary assessment of the method's value, but lap shear and peel data were also obtained. Some old data from a not too well controlled process, using old materials provided mild encouragement as to the merits of the process. Based on these results, a more controlled investigation was launched. Amchem supplied fresh materials.

A series of wedge specimens were prepared using both old and new solutions. Some of the specimens were primed and some unprimed. The nonchromium solution was also included and phosphoric acid anodized specimens were prepared as controls. The results of this work were inconclusive, largely because the older alodine solutions gave the best results - comparable in fact to the primed and phosphoric acid anodized specimens. The nonchromium treated materials did not appear promising.

Data from lap shear and floating roller peel work also presented anomalies. Lap shear strengths were surprisingly low at high temperatures. Peel strengths were low at room temperature but increased at the higher temperatures.

This program has been under review for some time. The data seems both encouraging and discouraging, the nonchromium materials have not been promising, and the presence of hexavalent chromium and cyanides remain as serious concerns.

5. Inorganic Primer

The current state-of-the-art adhesive bonding process for aerospace applications involves the use of corrosion inhibiting adhesive primers to impart long-term environmental durability. The best of these primers contain chromate compounds which present environmental disposal problems.

In order to eliminate the disposal problem encountered with the use of these primers, United Technologies Research Center investigated the use of an inorganic primer based on hydrolyzed metal alkoxides. They demonstrated that when these primers were applied to aluminum surfaces that had been prepared with standard acid etch (FPL, Pasajel, or SmutGo) and anodization (PAA) treatments, equivalent or superior durability (based on wedge crack propagation behavior) to chromate containing corrosion inhibiting primers could be achieved. Studies by UTRC indicated that thicker coats of inorganic primer (up to ~4500A) and longer hydrolysis times provided superior performance. Both of these variables were included in this study.

The objective of this investigation was to evaluate the inorganic primer on a nonacid surface preparation which could be used in field repair. The evaluation consisted of the preparation and testing of wedge crack specimens in accordance with ASTM D3762. The inorganic primer solution consists of a 1% solution of E-8385 (sec-butyl) aluminum alkoxide (from Stauffer) in toluene.

In addition to the inorganic primer, BR127 (a chromate containing primer) and a silane primer (X1-6100) being studied independently at WRDC/MLSE were included in the study for comparison purposes.

The surface preparations used in this study were phosphoric acid anodization as a baseline and both dry and wet abrasion processes typically used in repair. All of the specimens were bonded with EA9628 adhesive. This adhesive was known to fail cohesively in a wedge test specimen with a good surface preparation and primer but durable enough to force adhesive failures with a nondurable surface preparation and primer.

The results of the crack growth tests performed in this study are presented in Table 32. It is evident from the data in Table 32 that none of the combinations of surface treatment and primer that were tested were as good as the state-of-the-art PAA/BR-127 combination. The effect of the several processing parameters are discussed next.

- Wet vs. Dry Scotchbrite Abrasion (Cases 5 & 6 vs. 8 & 9)
  - There did not appear to be a significant difference between these two surface preparation techniques. Both gave comparable crack growths for similar conditions although the Wet method was very slightly better.
- 20 vs. 60 minutes Hydrolysis Time (Cases 5 & 8 vs. 6 & 9)
  - There did not appear to be a significant difference between the two hydrolysis times although the 20-minute time was very slightly better.
- 3 Coats of 1% Solution vs. 1 Coat of 3% Solution (Case 6 vs. 7)
  - One coat of 3% solution appears to be significantly better than three coats of 1% solution although it is still significantly inferior to the PAA/BR-127 performance.
- X1-6100 vs. Inorganic Primer (Case 10 vs. 5-9)
  - The silane primer (X1-6100) appears to be significantly better than the inorganic primer (E8385) although it is still significantly inferior to the PAA/BR-127 performance.
- PAA vs. Scotchbrite Abrasion (Case 3 vs. 5 & 8)
  - When primed with the inorganic primer, a surface prepared by PAA gives significantly better performance than either dry or wet SBA.

These results appear to be consistent with results at United Technologies reported by telephone. Results of their tests with the inorganic primer on mechanically abraded surfaces were not as good as they were on acid etched or anodized surfaces.

6. Cast Aluminum Bonding Study

High quality aluminum alloy castings are finding application on Air Force weapon systems owing to the significant cost savings over those components machined from plate stock. However, it is not known if durable adhesively bonded joints can be fabricated using present bonding technology with aluminum castings. This effort is intended to determine the feasibility of adhesively bonding aluminum castings using the same surface preparation

techniques developed for plate aluminum. Data are presented for the long-term durability, effects of temperature, and environmental exposure of those bonds.

It would have been desirable to obtain flat cast aluminum sheet having similar thickness as sheet aluminum normally used to evaluate adhesives and adherend surface preparations. Foundries who are capable of casting aluminum were contacted, but do not ordinarily cast such sheet. The cost of obtaining specially cast aluminum would have been prohibitive. The WRDC/MLSE project engineer located sections of a large cast aluminum aircraft bulkhead from another Air Force sponsored program which was available and suitable for this investigation.

Cast Aluminum Bulkhead:

Test specimens were obtained from pieces of the Station 170 cast aluminum bulkhead of a YC-14 fuselage. The bulkhead was cast A357 aluminum alloy. A357 is an age hardenable aluminum-silicon-magnesium alloy characterized by excellent castability, good response to heat treatment, high resistance to corrosion, and good weldability. The chemical composition of aluminum alloy A357 is shown in Table 33, and the heat treatment used is shown in Table 34. Details of the Air Force sponsored program to develop the bulkhead, conducted by the Boeing Company, are discussed in AFFDL-TR-78-62, "Cast Aluminum Structures Technology (CAST) Manufacturing Methods."

Adhesives:

Two adhesive types were used in this investigation. One, a 250°F curing system, was Hysol's EA9628. Two forms of the EA9628 were used; EA9628H has a nylon scrim and weighs 0.080 lbs/ft^2, and EA9628NW has a nonwoven mat scrim and weighs 0.060 lbs/ft^2. The original intent was not to use two forms of the adhesive. This was discovered after the fact and after examination of the data it was felt that it had little effect upon the results of this

investigation. The second adhesive type was a 350°F curing system, American Cyanamid's FM-300.

All specimens were primed with BR-127 corrosion inhibiting primer. The thickness of primer was difficult to control owing to the roughness of the aluminum casting, but we believed it was close to that usually recommended, 0.0002 inch.

Test Specimen Machining:

The cast aluminum bulkhead was difficult to work with owing to the size, roughness, and location of large ribs. The ribs were about 1/8 inch high and wide and ranged from 5/8 inch to 7/8 inch apart. There were a few areas free of ribs, and these areas provided pieces for wedge crack test adherends.

Once the flat sections between the ribs were cut out, these sections, usually about 3 inches by 10 inches, were cut into smaller pieces very close to the required final size and then machined to the specified dimensions on a milling machine.

Some of the finished specimens had small bumps or other extrusions on the surface. These were either milled clean or ground off with a manual die grinder. All lap shear bonds were bonded on the as-received rough casting surface. The wedge crack specimens were bonded on the as-received rough casting surface or areas with minimum grinding. Usually, these ground areas were restricted to the side opposite the bonding surface.

Surface Preparation:

Surface preparation procedures used in this study are standard techniques that represent current aluminum bonding technology. The process listed below was used for both the cast aluminum and 2024-T3 aluminum adherends.

Adherend Etch Procedure:

1. Solvent wipe with acetone.
2. Vapor degrease for 10 minutes in trichloroethane.

3. Alkaline wash for 10 minutes at 155 ± 5°F (Note 1).

4. Water rinse for 10 minutes in a continuous flow tap water bath.

5. Deoxidize in optimized FPL (OFPL) etch solution for 10 minutes at 155 ± 5°F (Note 2).

6. Water rinse for 10 minutes in an agitated continuous flow tap water bath.

7. Anodize for 20 minutes in a 9 to 12 percent by weight phosphoric acid anodize solution per ASTM D3933 at 15 ± 1 volts (Note 3).

8. Water rinse for 10 minutes in a continuous flow tap water bath.

9. Force dry with a heat gun or in an oven for 10 minutes at 150°F.

NOTES:

1. Alkaline solution:
   a. 1 gallon tap water
   b. 170 grams Turco 4215
   c. 7 ml Turco 4215 additive

2. OFPL etch solution:
   a. 11.1 liters tap water
   b. 417 grams sodium dichromate ($Na_2Cr \cdot 2 H_2O$)
   c. 2 liters sulfuric acid (reagent grade)
   d. 26 grams shredded 2024-T3 aluminum

3. Phosphoric acid anodize solution:
   a. 1 liter tap water
   b. 69 ml phosphoric acid, 85 percent
      or
      84.5 ml phosphoric acid, 75 percent

SEM Investigation:

Samples from both types of adherends, lap shear and wedge crack growth, were submitted for scanning electron microscope (SEM) evaluation. Cast aluminum samples were viewed at 100X and 1000X magnification. The porosity of the castings was apparent, making it difficult to detect any anodization.

Primer Application:

The primer, American Cyanamid BR-127, was applied with either a spray gun or an air brush. The primer was applied in several passes rather than a one- or two-pass buildup. After spraying, the adherends were air dried at room temperature for one-half hour and then dried in an oven at 250°F for an hour. The primed adherends were covered with clean, lint-free tissues and all panels were bonded within 24 hours of adherend priming.

Bonding Procedures:

The 2024-T3 specimens were bonded in a press using the standard procedure: apply 30 psi, heat to the required temperature (250°F or 350°F depending on the system), and cool. Because they were bonded in panel form, these panels were easy to lay up using standard fixturing.

The cast aluminum specimens were layed up individually. The film adhesive was applied, then binder clips were used to apply pressure to the bond area and keep the adherends from slipping. Lap shear and wedge crack specimens were layed up the same except more binder clips were used for wedge crack. The specimens were cured in an oven at either 250°F or 350°F, depending on the adhesive specimens.

Tensile Lap Shear and Wedge Crack Testing:

Lap shear tests were conducted similar to ASTM D1002. Specimens made with 2024-T3 aluminum were the standard (fully machined after bonding) type of test panel. The casting specimens did not meet the D1002 specification exactly. They were 0.475 inch wide and of slightly varying thicknesses, in the neighborhood of 1/8 inch. We attempted to match the thicknesses of both adherends used in a specimen.

Due to the roughness of the cast specimens, it was difficult to determine the applied primer thickness. The method used to estimate the thickness was to compare the color of the primer on a machined portion of the adherend to a pair of model panels that

were coated with the minimum and maximum allowable primer thickness.

As indicated earlier, an objective of this study was to determine the effects of temperature and environmental exposure on adhesively bonded joints using aluminum castings. Table 35 outlines the test matrix.

Wedge crack tests were conducted similar to ASTM D3762. All specimens were the specified size, although the 2024-T3 specimens were bonded as a panel and machined into individual specimens, while the cast aluminum specimens were bonded individually. The test matrix used in this portion of the study is outlined in Table 36.

Discussion of Results:

The goals of this study should be reviewed before discussing results. First and foremost, the testing and evaluation of the surface preparation for aluminum castings was to be studied. Other factors to be determined included (a) the effects of temperature and humidity on lap shear strength, (b) determining the effects of salt spray and humidity on crack growth, and (c) comparing values gathered from cast aluminum adherends to values obtained using 2024-T3 aluminum.

Lap shear specimens were prepared and tested as described earlier. The lap shear results are presented in Table 37 and represent an average of five test specimens for each condition. From the limited amount of data obtained, tensile lap shear strengths are slightly lower, but satisfactory, when cast aluminum adherends are compared to 2024-T3 adherends. The failure mode was cohesive for both types of adherends.

Wedge crack growth specimens were prepared and tested as described earlier. The wedge crack growth results are presented in Tables 38 and 39 and represent an average of five test specimens for each condition. Crack growth data, which has generally proven to be a very successful method of evaluating surface preparation, appears to be very similar when comparing

cast aluminum and 2024-T3 aluminum. The failure mode was cohesive for both types of adherend materials.

From the data obtained for tensile lap shear and wedge crack growth, it can be said that the surface preparations generally used for plate aluminum can be used for cast aluminum.

7. High Temperature Adhesives

With the development of new air-to-air missile technology, which may utilize graphite/polyimide composites as body and/or control surfaces, it was discovered there was a lack of data available on structural adhesive systems suitable for use at temperatures in excess of 538°C (1000°F). A continuing effort to build a high temperature adhesive data base has been conducted. In an earlier effort, data were generated on several adhesives including LR-600, LaRC-13, PMR-15, FM-34, FM-34B-18, FM-36, and PBI. The effort described here expanded upon that earlier data base. A limited amount of data was generated on NR-150, IP-600, and FA-7001 and is shown in Tables 40 and 41.

8. B1-B Repair Resin

A screening program was completed in 1988 which compared various wet-layup epoxy resin systems as candidate materials for repair applications on the B1-B. A two-part resin, designated EA9396 and manufactured by Hysol, was selected at the conclusion of these screening tests as the material to be more thoroughly characterized for design allowable data.

Three batches of this material were obtained. Specimens were prepared and tested in accordance with the test matrix listed in Table 42 using both E-glass and graphite fabric reinforcements. All of the laminates prepared and machined into specimens were vacuum-bag-cured at 200°F for 45 minutes except for those in Task 16. While there were a few minor deviations from the work plan outlined in Table 42, the data generated during this program

generally corresponds to that listed in Table 42. The data generated during this program are presented in Tables 43-69. Figures 12-21 supplement these data.

The data in Tables 43 and 44 relate to the degree of cure that can be achieved for various time/temperature histories. Table 43 illustrates the results of calorimetric measurements of the heat released during the exothermic cure reaction. As would be expected, the higher the cure temperature, the shorter the period of time needed to complete the curing reaction and the higher the degree of cure that is achieved. The data in this table also illustrate that little or no additional curing occurs at room temperature after an elevated temperature cure has been completed. Figure 12 illustrates the type of DSC curves obtained for the isothermal tests listed in Table 43. Part (a) illustrates the isothermal test, while part (b) illustrates the dynamic DSC test from which residual exotherm is obtained. Table 44 illustrates the effect of time/temperature cure history on a resin/interface dependent mechanical property. In general, the data in Table 44 corroborates the calorimetric data in Table 43 in that the property levels decrease with decreasing cure temperature. Figures 13-15 present FTIR spectra obtained from EA9396 at various stages during cure/postcure. While substantial changes are evident and are to be expected between the uncured and cured states, there are also very noticeable differences between the cured and postcured states.

Tables 45-55 present the tensile, compressive, inplane and interlaminar shear properties measured at various test conditions on the three different batches of resin. In general, the graphite-reinforced laminates retained a higher fraction of their room temperature dry property levels than the glass-reinforced laminates after wet-aging. In addition, the fiber-dependent tensile properties exhibited substantially less degradation with increasing test temperature than the matrix/interface dependent compressive and shear properties. The matrix/interface dependent properties fell off more with increasing temperature for the wet-aged condition than for the dry condition. The decrease in wet-aged property levels is particularly marked for the case of the

glass-reinforced tensile data. The glass fabric used to generate these data had a Volan-A sizing. This is supposed to be epoxy-compatible and environmentally resistant. After obtaining these data, some A-1100 sized glass fabric was obtained and comparison panels prepared for tension, inplane shear and interlaminar shear testing. These data are presented and discussed later.

Table 56 illustrates the effect of simulated 350°F postcure cycles on interlaminar shear strength. It is evident that essentially no degradation is observed as a result of exposure to the 350°F temperature cycles. Table 57 presents the results of bearing strength tests.

Table 58 illustrates the effect of wet-aging on glass-transition temperature of EA9396 resin. While a substantial decrease is apparent from the dry to the wet test condition, the wet T_g value is still above the boiling point of water and still above the maximum test temperature employed in this program.

Table 59 presents the results of interlaminar shear tests on panels of varying resin/fiber content. While an effort was made to fabricate panels having a larger variation in resin and fiber content than that extant in this table, the innate nature of the fabrication process reduced the achievable variation to that listed in Table 59. On the plus side, this indicates that the process will produce reasonably repeatable laminate quality regardless of the resin/reinforcement ratio used during layup. As evident from the shear strength values listed in Table 59, there is relatively little effect of resin/fiber content on shear strength.

Tables 60-63 illustrate the effect of extended storage time on various resin characteristics. There is very little effect of storage time up to 12 months or at elevated temperature on viscosity, calorimetric cure behavior or shear strength. The only noticeable effect of storage on viscosity cure profiles is that it took longer to reach minimum viscosity after 12 months storage than it did for up to 6 months storage. Figures 16 and 17 present viscosity profiles for fresh resin and for resin which has been

stored at 120°F for 12 months. There is very little difference apparent. Figures 18-21 illustrate FTIR spectra for fresh resin (parts A and B) and for resin (parts A and B) which had been stored at 120°F for 12 months. The noticeable differences are that some of the absorption bands have diminished with storage. It is not possible at this time to attribute these bands to specific chemical groups or reactions.

Pot life/work life studies were carried out on both bulk resin batches and on wet-layup laminates. Tables 64 and 65 summarize the results of these tests.

The effect of vacuum level during cure was evaluated. Table 66 presents the results of these tests. It can be observed from these data that as vacuum level increases, greater compaction results. This is manifested in the progressively higher specific gravities and thinner ply thicknesses. No consistent relationship between cure vacuum level and shear strength is apparent.

Tables 67-69 present the results of the tensile, inplane and interlaminar shear tests on the specimens prepared with A1100-sized glass fabric when wet test data becomes available.

A technical report describing the details of the fabrication and testing procedures employed during this program as well as a thorough discussion of the results is being prepared.

9. Composite Paint Removal

This is a program of long standing that is nearing successful completion. The goal is to investigate the use of small plastic beads, of given size, shape, and hardness, fired at given angles and pressures to strip painted composite surfaces. The advantage is the removal of paint without liquid waste products. Much effort has gone into investigating this technique. A spectrum of mechanical properties--tensile, compression, flex, shear--are being determined before and after stripping to determine if the impacting plastic beads cause mechanical damage. These property determinations are done after one cycle of

painting/stripping, and after four cycles. NDE and SEM inspections are also included.

Several hundred specimens have been mechanically tested in the combined first phase (one painting/stripping cycle) and second phase (four painting/stripping cycles) of this program.

A paper has been completed for the Spring, 1990, SAMPE meeting detailing the effort of the first phase. The data for the second phase is currently undergoing evaluation. Several preliminary discussions have been held regarding the continuance of this effort using different stripping media, techniques, lay-ups, etc.

10. Thermoplastic Prepregging

This effort is an off-shoot of the Induction Heating program discussed later. It is relatively new and most of the work to date has consisted of equipment design and materials acquisition. The goal is to utilize a fluidized bed of thermoplastic (or aluminum) powder to impregnate a wet and tacky graphite fiber tow. The tow/matrix combination will be collected on a drum winder, cut into 4-inch x 4-inch lay-ups and consolidated under vacuum bagging and induction heating. Most of the effort to date has focused on the use of aluminum powder and a wet graphite tow (water base solution). These early runs have suggested technique and process improvements which are underway.

11. RMX Evaluation

This is a patch technique which was evaluated for the field repair of aluminum honeycomb structures. Once the damaged area is cleaned out, an oversized aluminum sheet is used with a resin/fabric underlayer to provide stiffness and fill the region of removed core. The aluminum sheet/core fill patch can be contoured to cover the hole and is then bonded, adhesively or by fastener, in place.

The patch application technique was not an issue. The question was whether the applied patch would restore sufficient integrity to be useful. Evaluation consisted of compression,

tension, and shear tests on both the skin and the core. The results indicated that the patch is useful for skin repair only. Our work did not indicate any load transfer from the surrounding core into the underlayer of the patch.

12. Heat Lamp Evaluation I

The goal of this program was to explore the possibility of using a 110V/250 watt heat lamp to dry out materials prior to initiating field repair. A frequent, if not omnipresent, problem in field repair work is absorbed moisture. It was hoped that this type heat source would be effective in drying the materials without damage. Various plastic laminates, honeycombs, and skins were used as experimental subjects. The position, distance, and angle of the lamp was varied, and a pattern of thermocouples was used to monitor temperature distribution profiles.

A computer program was generated to provide mapping of isotherms from a given set of experimental parameters. Examples of these isotherm maps are shown in Figures 22-24.

13. Heat Lamp Evaluation II

The success of the earlier Heat Lamp Evaluation program spurred interest to continue with a more closely defined effort. Lamp-to-panel distances were correlated with panel temperature profiles. The panel material was varied and dual lamp assemblies were also included. Once again isotherm maps were generated for each panel surface for given test parameters.

While these two Heat Lamp Evaluation programs provided a substantial amount of data, the use of heating lamps for this purpose has some drawbacks. The area of temperature rise is limited and nonuniform, the breakability of the glass lamp bulbs is a consideration, and the necessity for no blockage of the light path is limiting. In general, for field repairs, the heating blanket is preferred.

14. Nonautoclave I-Beam Fabrication

This program undertook an exploratory look at a difficult problem. The goal was to construct a composite I-Beam which could be loaded to simultaneous failure in three modes--tension, compression, and shear. Several units were fabricated and tested. The beams were 24 inches long x 3 inches high with 3/4-1 inch spars. Different resins were employed, with B-staging and curing variations, and a double vacuum bagging technique was used for consolidation. The design and confirmation of a curing tool was also completed. Nondestructive inspection indicated void problems in the web area. This was not a long-term program and in the summarizing description of the USAF engineer it was "a difficult undertaking with limited success."

15. Resin Transfer Molding

A small mold was designed and constructed to make approximately 1/8-inch x 11-inch x 11-inch panels to study the resin transfer molding (RTM) process. With six plies of woven fiberglass, a fiber volume of 51% was obtained using Dow Tactix 123/1431 epoxy resin. The mechanical properties (flexure strength and modulus and short beam shear, Ref. Table 70) were similar to those obtained with press cured panels although neither process was optimized to duplicate the higher pressures that are usually obtained in some production processes. Void content on both sets of panels ranged from 3.7 to 8.6%.

The RTM process was limited to a 60-psi pressurized resin supply but this was adequate to make representative panels. The point of resin introduction was changed from the edge of the panel to the middle to reduce air entrapment. The mold was preheated to 200°F to lower the viscosity of the resin for better flow and less porosity. A higher temperature would have caused gelation of the resin before filling of the mold cavity was completed.

16. Resin Transfer Molded Pressure Vessels

The objective of this task was to determine the feasibility of using resin transfer molding for filament wound pressure vessels. It was hoped vessels having much lower void contents than those fabricated using conventional wet winding techniques could be achieved. Pressure vessels were wet wound using conventional techniques, or dry wound and shipped to Radius Engineering and Tooling, Inc., Salt Lake City, Utah. Radius Engineering then applied the resin using transfer molding techniques. UDRI hydroburst tested the pressure vessels then dissected them and determined physical properties. Photo-micrographs of the wall cross sections were also obtained.

Mandrel Fabrication:

Several types of mandrel materials and fabrication procedures have successfully been used in the past. In retrospect the one which might have been most satisfactory for this project would have been a sand mandrel. Sand mandrels are cast using sand and a water soluble binder, such as PVA or sodium silicate. These mandrels are solid castings on a steel shaft and have excellent compression strength.

Another type of mandrel is one using water soluble plaster. This was the one chosen for this project. A plaster key is cast on the steel shaft and cardboard forms are used for ribs which stiffen the mandrel and form a cavity. The cavity helps lighten the mandrel and reduces the amount of plaster which must be dissolved later. Additional plaster is then screeded in place.

The reason for selecting the plaster mandrel was that with the materials and molds in-house, it was felt that a higher quality mandrel could be fabricated, at least in the time given to complete the task. Given more time it would have been desirable to fabricate a mold which would have been used for sand/sodium silicate mandrels. These would have had much higher compression strengths, which would have been an aid in the resin transfer molding operation.

Pressure Vessel Liner:

Once the plaster mandrel was fabricated and dried, an elastomeric liner was applied. This liner is required for pressure retention in hydroburst tests. Without it, the vessel would leak under pressure. Not much time was available for selection of a liner material. Advice was sought from personnel knowledgeable in elastomers, and a fuel tank sealant from 3M was selected, ED776. Multiple layers of the sealant were brush coated to build sufficient thickness for hydroburst.

Pressure Vessel Fabrication:

The pressure vessel selected was one nominally 6-inch as in diameter conforming to ASTM D2585. The carbon fiber selected was Hercules IM-6/12K tow. There was no particular reason for selecting this fiber other than the fact that it was in inventory at UDRI and was suitable for wet winding. The epoxy resin selected was from Shell Chemical, Epon 9405/9470. This resin was developed for both "wet" filament winding and resin transfer molding and therefore it was suitable for this task. The winding pattern selected was one with two polar 12.5° plies and four hoop plies. The bottle internal burst pressure should be about 3000 psi.

UDRI wet wound and fully cured a pressure vessel and delivered it to Radius Engineering to fabricate an arc-sprayed metal-faced composite mold (Arctool^{TM}). The fully cured bottle was used as a pattern in the mold process. Two "dry" wound bottles were then fabricated and delivered to Radius Tool for impregnation by resin transfer molding, using the mold patterned from the fully cured wet wound bottle. The "dry" wound bottles were actually wound using a very dilute solution of a wetting agent and a water soluble resin in water. This solution was used to prevent fiber damage during filament winding. The bottles were oven dried to remove the solution. All wound pressure vessels, both dry and wet, were wound with 7 lbs. tension on the 12K tow measured at the pay-off eye.

From a summary report supplied by Radius Engineering, impregnation of the first bottle was only partially successful. This was due to a bladder failure in the tooling during impregnation, and collapse of the inner plaster mandrel. A sand mandrel might have, at least in part, prevented the collapse. Impregnation of the second bottle was successful. Radius Engineering changed the tooling approach to prevent plaster mandrel collapse. Also, this new approach was less sensitive to variation in bottle dimensions. This successfully resin transfer molded bottle was shipped back to UDRI for hydroburst testing.

UDRI then "wet" wound and fully cured three additional pressure vessels using the same materials, winding pattern, and number of turns as the bottle which had been resin transfer molded. The plaster mandrels were then removed from all four pressure vessels. Although the particular plaster used for the mandrels is water soluble, it is a time consuming task and requires very hot water.

Hydrostatic Burst Testing:

The burst strengths of the "wet" filament wound pressure vessels were determined using a maximum 30,000 psi Haskel Hydroburst Tester. The test procedure was similar to that recommended in ASTM D2585. A typical wet wound pressure vessel before hydroburst is illustrated in Figure 25. The first attempt to hydroburst the bottles failed due to excessive leakage. This is usually caused by too thin of a liner. When the leakage is excessive, the hydroburst chamber will not pump enough water to pressurize the bottle because of water leakage.

The bottles were removed from the hydroburst chamber, drained of all water, and oven dried. The EC776 sealant viscosity is low enough so that material could be poured into the bottle and "sloshed" around until the solvent evaporated, leaving a thick coat of elastomer. This was repeated to insure that the liner was thick enough to hold the water during pressurization.

The wet wound pressure bottles were then successfully hydroburst tested. A typical wet wound bottle after hydroburst can be seen in Figure 26. The burst pressure was measured for two of the three wet wound bottles and the results are shown in Table 71. The third wet wound bottle was burst tested but the pressure was not recorded. It appeared to be in the same range as the first two. The results are lower than those predicted.

Several attempts were made to burst the resin transfer molded pressure bottle. The first two attempts were aborted due to excessive leakage. Slosh liners were applied using the same technique as for the wet wound bottles. During another attempt, some of the hoop fibers began to fail and leakage once again became excessive. Once again a slosh liner was applied and a final attempt to burst was conducted. During this attempt, a significant portion of a tow in the dome area fractured, and the leakage became so great that all hope to finally burst the bottle was abandoned. Figure 27 illustrates the RTM pressure bottle after attempting to burst test. Some loose hoop fibers can be seen. Figure 28 shows that the initiation of failure in the hoop fibers resulted from small wrinkles in the bottle, probably resulting from RTM processing. The large section of a tow in the dome area which fractured can be seen in Figure 29. At this time, it was suggested that during resin transfer molding, the resin only penetrated the outer plies and that the inner plies remained dry. This turned out to be partially true.

Physical Properties:

In order to verify that resin may not have penetrated the inner plies in the resin transfer molded pressure vessel, the bottle was dissected for physical examination. Actually it was intended that all bottles be dissected for examination. Pieces were cut from the dome area and the hoop area of each bottle. The liner was removed and the density, % fiber, % resin, and void contents were determined. Photomicrographs were also obtained.

When removing the liner from the resin transfer molded bottle, it was observed that some of the inner ply was indeed dry. The physical properties were then measured and are shown in Table 72. The first significant observation is that the measured void content in the resin transfer molded bottle is "0%". Reducing the void content in wound pressure vessels was one of the objectives and was obviously met. Also noted in the physical properties is that the resin content is lower in the resin transfer molded bottle, resulting in a high fiber volume. This is not to say that the resin content is too low and fiber volume is too high, but only that they are different than those obtained by wet winding.

Sections of both wet wound bottles and resin transfer molded bottles were mounted and polished for photomicrographs. Attempting to polish the pieces from the resin transfer molded bottle has led to a delay in completing this project. The elastomeric liner remained on the pieces to be polished. It was hoped that the photomicrographs would show the inside plies which were thought to be dry or resin starved. Polishing the sample with the soft elastomeric liner, with unimpregnated or at least partially impregnated plies which are also soft, and with the outside impregnated plies which are hard, turned out to be impossible. Photomicrographs were taken in the dome area and the hoop area but not adjacent to the inner plies or the elastomeric liner.

The photomicrographs do verify that at least in the impregnated areas, the resin transfer molded bottle does appear to be void free in both the dome and hoop areas. The photomicrographs also verify that the wet wound bottles have some voids typical for this fabrication procedure. Figures 30 and 31 illustrate the voids in the hoop and dome areas in the wet wound bottles. The void-free areas in the hoops and dome for the resin transfer molded bottle are illustrated in Figures 32 and 33. Also, note in Figure 32 that the resin rich area is completely free of porosity. There were concerns that the choice of resin may have led to

porosity due to low vapor pressure additives. The resin rich area is caused by a gap between hoop windings.

The type of mandrel used here was a water soluble plaster. Any future work should be done with sand, which is stronger and stiffer. Although the sealant liner material used was satisfactory, a thicker layer should be applied. Other liner materials should also be investigated.

Even though the "dry" wound bottles were wound with a water solution, some gapping and roping was observed. Additional winding trials to eliminate this would be required.

The burst pressure for the wet wound bottles was lower than anticipated. The hoop plies in the resin transfer molded bottle began failing at a low pressure along what appeared to be a wrinkle. The inner plies in the resin transfer molded bottle appear to be dry.

The void content in the wet wound bottles appears to be what is normally expected. Photomicrographs and physical properties indicate that the resin transfer molded bottle is void free.

17. Evaluation of XP2942/310 Polymer

A program was conducted to evaluate the processing characteristics, and the physical and mechanical properties of a proposed high temperature polymer from Pyroite Polymer International (PPI) identified as XP2942/310. According to PPI, "A major advancement in polymer technology had been achieved in crosslinking an inorganic polymer with an organic polymer." The polymer was said to offer high temperature continuous service to 316°C (600°F) with the processability of epoxies.

Two batches of prepreg were received by UDRI from PPI. Upon receipt some physical property measurements were obtained. These included gel time, DMA, and DSC. The gel times obtained at UDRI on Batch No. 1 differed from that reported by PPI, but there was close agreement for Batch No. 2. PPI did not supply DMA or DSC data on either batch. DMA and DSC are often used as an aid in

determining cure schedules. There were considerable differences in the curves obtained between Batch Nos. 1 and 2.

The cure cycle used for panels fabricated from Batch Nos. 1 and 2 were different from each other and were supplied by PPI. These cure cycles are not necessarily those which would have been chosen by UDRI based upon the DMA and DSC data.

Physical properties were measured on the cured laminates. These included density, DMA, and DSC. PPI indicated that the T_g was 343°C (649°F) as measured by DSC. At UDRI there was no apparent T_g as measured by DSC on either batch. However, the T_g as measured by DMA was 170°C (338°F) for Batch No. 1. Batch No. 2 had three tansitions measured by DMA, 237°C (459°F), 343°C (649°F), and 365°C (687°F). The second corresponds to the T_g reported by PPI.

Flexure specimens were machined from the cured panels and tested in accordance to ASTM D790 at ambient and elevated temperature. Table 73 shows the data reported by PPI and that obtained at UDRI. There is a considerable difference between the data obtained by the two sources, in particular at elevated temperature. Also of importance is that the failure mode at elevated temperature is thermoplastic.

In summary the following observiations are noted:

(a) The prepreg material received from Batch Nos. 1 and 2 both handle and process similar to epoxies;

(b) The material appears to be limited to temperatures at or near the lower T_g;

(c) Batch No. 2 has a higher T_g and better elevated temperature properties than Batch No. 1; and

(d) The material appears thermoplastic above the lower T_g.

18. Panel Fabrication

At the request of the WRDC/MLSE, a graphite/epoxy prepreg, 3501-6/IM-6, has been kept in inventory to supply panels in support of various WRDC/MLSE projects. Nearly 100 panels have been fabricated. Thirty-two 2-ft. x 2-ft. x 16-ply panels were fabricated and delivered in support of the paint removal program. Thirteen panels were fabricated in support of programs concerning the use of metallic fasteners in composites. All of these panels were 2 ft. x 2 ft. and ranged from 16 to 72 plies. In addition, three panels, 1 ft. x 1 ft. x 24 ply, were fabricated and 12 holes were drilled in each for the addition of aircraft grade fasteners. Several trials were made before satisfactory holes were drilled. The fasteners were then attached, and the panels were delivered to the WRDC/MLSE.

Two 16-inch x 16-inch x 0.2-inch glass/epoxy panels were fabricated and delivered to MLSE. These panels were purposely fabricated with a low resin content to simulate the conditions in filament wound radomes. To obtain panels with low resin content, UDRI prepared the prepreg in-house. These panels were then impact tested and compared to resin transfer molded panels.

Twelve 1-ft. x 1-ft. x 14-ply graphite/polyimide panels, CPI 2237/PMR-15, were also fabricated and delivered to WRDC/MLSE. Physical properties were also determined for each panel. These panels were intended for drilling experiments.

Some graphite/thermoplastic panels were also fabricated and delivered to MLSE. Five graphite/PPS and five graphite/PEEK panels were fabricated.

19. Fabrication and Testing of Panel with Slip Plies

In some filament wound motor cases the hoop plies (90°) are wound with fibers which are coated with release agent. The reason for this is that as the vessel is pressurized, its length begins to grow and debonding and cracking begins in the hoop plies. This cracking can grow in an uncontrolled manner and lead to a

catastrophic failure. By coating the hoop fibers, a more controlled cracking and debonding situation exists. Also, less fiber damage occurs, and since the hoop stress is fiber dominated, a higher burst strength can be achieved. One potential problem may be that the release agent might migrate from the hoop plies to the helical plies (those running longitudinally along the vessel axis) which may cause premature failure during pressurization.

Filament wound pressure vessels, even on a small laboratory scale is expensive. Therefore, a flat panel was designed to represent both the helical and hoop plies and included a rubber pad which is usually used at the junction of the vessel and the skirt near the dome area. The skirt is used to mechanically fasten the motor case in the missile body. This area has high stress concentrations and often leads to premature failure.

The materials used were the same or similar to those often used in filament wound motor cases. The fiber was Kevlar 49 and the resin was an epoxy, Epon 9405. Fiber to be used in the simulated hoop plies was coated with Frekote 44, a release agent known to release from epoxies.

Since the panel was to simulate a wound structure, it contained 90° plies, representing the hoop plies, and $\pm$25° plies, simulating helical plies. These angles were chosen because they conform to the winding angles used on some motor cases using slip ply techniques. A length of fiber was wound on a mandrel with release agent in the resin bath. Heat was applied to the fiber immediately after coating to cure the Frekote 44. The amount of release agent picked up was 1.5%. Prepreg tapes were then wound onto the drum winder. The release agent coated fiber was wound into tapes for the hoop plies, and the tapes for helical plies were wound with uncoated fibers. The Epon 9405 epoxy resin system was a bisphenol A/epichlorohydren resin, with a non-MDA aromatic amine curing agent. The formulation was as follows:

- Epon 9405 resin - 100 parts by wt.
- Epon 9470 curing agent - 28 parts by wt.
- Epon 537 accelerator - 0.25 parts by wt.

After being impregnated, the prepreg was advanced on the drum winder by heating the material to 150°F by the drum's internal heaters. The temperature was maintained approximately 2 hours. The final resin content of the prepreg was 41.9% for the helical plies and 47.0% for the hoop plies.

The panel fabricated was 4-inches x 12-inches with the 12-inch direction in the 0° or longitudinal axis. The rubber pad was a polysulfide type compound conforming to MIL-S-8802. The pad was bonded on the panel with FM-73, and American Cyanamid adhesive.

The prepreg and adhesive were co-cured using the cure cycle of the Epon 9405 resin. The autoclave cure cycle is shown below.

- Apply full vacuum
- Heat to 250°F @ 3°F/min.
- Apply 85 psi as soon as 250°F is reached
- Hold @ 250°F for 1 hr.
- Heat to 300°F @ 3°F/min.
- Hold @ 300°F for 1 hr.
- Heat to 350°F @ 3°F/min.
- Hold @ 350°F for 1 hr.
- Cool to 100°F @ 5°F/min.
- Release vacuum and pressure

Two 1-inch x 12-inch specimens were machined for test. An edge piece was used for initial test setup. Loading tabs on the first sample were bonded on the end with Kapton in the hoop plies. The second specimen had the tabs bonded on the end with the Kapton in the helical plies. The tabs and the area under the tabs were abraded with sandpaper, solvent wiped, then the tabs were bonded on with Hysol 9320 room temperature curing adhesive.

The specimens were marked in 1/2-inch increments starting from the end of the Kapton. Two cantilever beam fracture tests were then performed at the following test speeds. Initial crack length was 1-1/4-inches.

Crack Length	Test Speed
1.25" → 2.25"	0.02"/min.
2.25" → 3.25"	0.05"/min.
3.25" → 4.25"	0.1"/min.
4.25" → 5.25"	0.2"/min.

After testing the specimens were delivered to WRDC/MLSE for fractographic study.

20. Aircraft Battle Damage Repair (ABDR) of Transparencies

Repair methods are currently being developed to provide increased sortie generation rates in a wartime environment. Recently, several concepts which could provide ABDR capability for aircraft transparencies have become available. The objective of this project was to evaluate these concepts in the laboratory and to develop an approach for deployment to field units.

Prior to this program, damaged transparencies were repaired by bolting a sheet of aluminum over the damaged area. This technique is sufficient to keep the windblast out of the cockpit but would be less than optimum for an aircraft making a combat mission. The primary objective was to evaluate induction heating as a repair technique. Polycarbonate, cast and stretched acrylic transparencies of various classes and construction types were considered.

The Inductron Corporation's TOROBONDER induction heater was used to fuse patches to polycarbonate, cast acrylic, and stretched acrylic canopies as well as to provide a heat source for structural bonding of patches to each type of transparency. Both polycarbonate and cast acrylic patches in thicknesses ranging from 1/16-inch to 3/8-inch were evaluated. Two patch sizes, large (4-inches x 6-inches) and small (3-inches O.D.), were evaluated. A heating blanket or hot-air gun was utilized to pre-form the patch to the canopy contour prior to bonding.

The evaluation of the TOROBONDER for heat fusing of patches to damaged transparencies included the parameters listed in Table 74.

The evaluation of the TOROBONDER for adhesive bonding of patches to damaged transparencies included the parameters listed in Table 75.

Fusion Bonding:

Experimentation included fusion bonding various patches to both flat samples as well as canopy surfaces of polycarbonate, cast acrylic, and stretched acrylic. This experimentation indicated that fusion bonding could be used successfully to apply a patch to both polycarbonate and cast acrylic surfaces. In addition, it was determined that the TOROBONDER could not be used to apply a patch to any type of stretched acrylic material because the temperatures necessary to effect melting of the stretched acrylic surface caused significant shrinkage and cavitation of the stretched acrylic substrate. The cavitated areas in the bond line between the patch and the stretched acrylic canopy leaked when subjected to the leak test criteria, although the patch adhered to the canopy at certain points in the bond line. The application of an RTV sealant to the cavitated areas of the patch resulted in a leakproof patch.

Adhesive Bonding:

Candidate adhesives compatible with transparency materials were selected for this project. These adhesives were evaluated for repair simplicity, shelf life, work life, surface preparation requirements, and equipment needed.

Summaries of experimental results obtained for each type of transparency and both types of induction heating methods (fusion bonding and adhesive bonding) are listed in Tables 76-78.

Conclusions and Recommendations:

Both fusion and adhesive bonding can be used successfully to accomplish ABDR. Many structural criteria as well as material and process considerations were evaluated in this work. Induction heating was utilized to accomplish both fusion bonding and adhesive bonding.

The following conclusions concerning fusion bonding are based on experimental results. This technique is better suited for smaller damage, but can successfully patch larger damaged areas with only a slight amount of difficulty. Although the difficulty increases slightly when fusing a large patch to a canopy substrate, less time is required to fuse a large patch than to adhesively bond a large patch using induction heating to cure the adhesive. In addition, induction heating results in the cavitation of stretched acrylic material but successfully fuses patches to all other canopy materials.

The following conclusions concerning the use of induction heating to adhesively bond canopies are based on experimental results. This technique is very good for small patches, but requires more time than fusion bonding when curing the adhesive for a large patch. The use of a heating blanket to cure the adhesive on a large patch required less time than induction heating. In addition, the use of induction heating to adhesively bond canopies requires the use of more equipment than fusion bonding. Adhesive bonding using the induction heater is viable on all canopy materials.

The use of the T-1000 TOROBONDER is not recommended for aircraft battle damage repair of canopies. Putting the unit in the ABDR kits for repair of canopies, and training the personnel to operate the equipment does not provide an advantage over the existing tech-order (TO) repairs which are primarily bolt-on patches. The current T-1000 induction heating equipment does not have the temperature control that is desired. However, the use of induction heating will be reevaluated after additional work on

structures and hydraulic tubing (under separate efforts) is completed since these efforts involve modifying the equipment to make it more field usable and controllable.

A more detailed description of the procedures used and results obtained on the Aircraft Battle Damage Repair of Transparencies project can be found in technical report WRDC-TR-89-4148.

21. Evaluation of Coated Polycarbonate

Five coated transparent polycarbonate samples from Epolin were received for optical testing, QUV exposure, and coating adhesion tests. Transmittance and haze of each sample was measured initially and after 1-, 2-, and 3-year equivalent QUV exposures. Initial visual appearance was recorded. The only visible change in the samples was a progressive yellowing with each year of aging. Table 79 presents the haze and transmittance measurements.

Tape adhesion tests were also carried out on these samples after 1, 2 and 3 years of QUV exposure. Table 80 presents these results.

22. Fuel Seals

The problem of fuel leakage is one of long standing and of obvious priority and concern. For the past several months, UDRI in collaboration with several USAF groups, have been addressing this problem, both from the perspective of elastomeric seal-fuel compatibility and, more recently, to include actual dynamic testing.

A presentation was given in September 1989, to the interested USAF parties detailing the compatibility and static testing results. There are two O-ring formulations in use: nitrile and fluorosilicones. These and two other base elastomers were studied in four fuels (three JP-4 modifications and JP-8) at three temperatures (-65°F, R.T., and 140°F). A substantial amount of data was generated to provide fundamental information on the static

performance of these elastomers and fuels under a variety of conditions. Soxhlet extraction data were also obtained and JP-7, and modifications thereof, has since been added to the program.

Several major conclusions have become evident thus far. (1) The volatility of JP-4 will always be a concern. In part, this volatility is due to the presence of low molecular weight aromatics, but their concentration varies between 2 and 15% (always within allowable Spec). These components also play a key role in volume swell especially with the nitrile material. (2) The range of low MW aromatics in JP-8 is consistently less than 1%. (3) There is a marked volume swell difference between JP-4 (higher) and JP-8 for fluorosilicone rubber. (4) Soxhlet extraction data indicated that the fuels remove the plasticizer from nitrile but have little chemical effect on the other materials. Overall these data suggested different modes of failure for nitrile (loss of plasticizer) and fluorosilicone (low volume swell, at least in JP-8).

Planning has continued to develop a dynamic testing phase wherein actual Wiggins couplers in different fuels under a variety of temperatures and pressures will be investigated. The goal of this work is two-fold. There is the problem itself to address and resolve, and second, there is the desire to develop a test fluid and test procedure that will serve as a qualification practice in the future.

23. ARM-100 Lubricating Oil

This is a new lubricating oil available from the SAE. The goal was to evaluate seals in the fluid at temperatures matching the performance levels of the elastomers. Volume swell was the key parameter of interest and the data in Table 81 summarize the results of this program. All agings were for 70 hours. The change in hardness along with some of the volume swell levels show the effects of the fluid.

24. Fabrication of Plastic Specimens

This was an effort for Hq/AFLC in which four acrylic specimens, 6-inches x 20-inches, were cut, formed, and drilled for use as teaching aids.

25. Meg-A-Temps Insulation

This consisted of the evaluation of a surface insulation for aluminum. A box-like test fixture was constructed of particle board and aluminum wherein the aluminum wall could be insulated and thermocoupled. Tests were conducted at temperatures from room temperature to 250°F with the aluminum bare, Meg-A-Temp insulated, and fiberglass insulated. The Meg-A-Temp insulation proved inferior to fiberglass.

26. Canopy Patching

The goal of this program was to investigate the possibility of using an O-ring sealed plug as a canopy patch. Transparent EDPM O-rings were used in a carefully machined plug, gland, and hole assembly. Several combinations of dimensions were studied but none could withstand the atmospheric pressure differential.

27. Photographic/General Support

General support was provided for photographing, slide making and general display preparation for briefing purposes.

28. PI Radome Thermoplastic Potential

A PI/quartz composite sample was studied to determine its thermoplastic performance, specifically to see if it would respond to repair (self-bonding) at 700°F/200 psi under nitrogen. The part was held under these conditions for 20 minutes, but no evidence of bonding was observed.

29. Subcontracted Effort

A subcontract was let to the Inductron Corporation to develop and deliver induction heating units for use with "memory metal" couplings for hydraulic tubing repair. Using a small, portable power supply, Inductron had demonstrated the ability to inductively heat small localized areas and to custom design and build special heating probes to heat various shape and size components. The goal of this subcontract was to develop a system to heat shrink memory metal couplings so that in situ repair of hydraulic tubing could be accomplished. Inductron investigated and evaluated the possibility of using a single heating probe for several coupling diameters and then fabricated and delivered the probes necessary to accommodate couplings ranging in diameter from 1/4 to 1-inch. A total of three probes were found to be necessary and were fabricated and delivered. One accommodated 1/4, 3/8, and 1/2-inch couplings. A second accommodated 5/8 and 3/4-inch couplings and the third accommodated 7/8 and 1 inch couplings. In addition, a set of instructions for using the heating probes with the power supply was prepared and delivered. All these deliverable items were forwarded to WRDC/MLSE.

TABLE 1

MATERIAL FORM AND DESCRIPTION AS REQUIRED IN FMS-1013C

Identification	Weight	Description	Source
Adhesive Form 1B	0.14 to 0.16 lb/sq.ft.	Film, 18-20 mils	RB398NA, Reliable
Primer Form 2	12 ± 2% solids	Adhesive Primer	R-500, Ciba Geigy
Adhesive Form 3&4	0.085 to 0.10 lb/sq.ft.	Film, 14-17 mils	AF-130/2, 3M
Additional Material Requirements			
Aluminum Sheet		2024-T81	Midwest Metals
Glass/Epoxy Prepreg		RN 1601	Reliable
Honeycomb Core		1/8 5052/8.1	Hexcel
Honeycomb Core		HRP-3/16-7	Hexcel
Adhesive Primer	30%	35 G/L VOC	EA-9289, Hysol
Adhesive Primer[1]	20%	58 G/L VOC	EC-3983, 3M

NOTE: 1. Used for tests in Task Nos. 15 and 16 only.

TABLE 2

QUALIFICATION TEST REQUIREMENTS

No.	Adhesive Form	Property	Test Panel Para.	Test Method FPS-1028 (Unless Noted)	Number of Specimens	Test/Temp/ Condition(°F)	Minimum Required Average
1	1B	Laminate-to-aluminum Lap Shear	9.5	A-054	10(1)	-65	1350 psi
					10(1)	75	1600 psi
					10(1)	270	1100 psi
					10(1)	350	1050 psi
2	1B	Aluminum Overlap Shear Strength	9.6	MMM-A-132	10(1)	-65	2100 psi
					10(1)	75	2300 psi
					10(1)	270	2300 psi
					10(1)	350	1800 psi
3	1B	Aluminum Overlap Shear Creep	9.6	MMM-A-132	5(5)	75	0.015-inch, max.
					5(5)	270	0.015-inch, max.
					5(5)	350	0.015-inch, max.
4	1B	Sandwich Flatwise Tension	9.2	B-057	6(1)	-65	3375 lb.
					6(1)	75	3475 lb.
					6(1)	270	2900 lb.
					6(1)	350	2100 lb.
5	1B	Short Beam Sandwich Shear	9.3	B-053	3(2)	-65	2275 lb.
					3(2)	75	2275 lb.
					3(2)	270	2190 lb.
					3(2)	350	2000 lb.
					3(2)	400	1720 lb.

TABLE 2 (Continued)

QUALIFICATION TEST REQUIREMENTS

No.	Adhesive Form	Property	Test Panel Para.	Test Method FPS-1028 (Unless Noted)	Number of Specimens	Test/Temp/ Condition (°F)	Minimum Required Average
6	1B	Long Beam Sandwich Shear Strength	9.3	B-060	3(2)	-65	2275 lb.
					3(2)	75	2275 lb.
					3(2)	270	1800 lb.
					3(2)	350	1625 lb.
					3(2)	400	1475 lb.
7	1B	Sandwich Beam Creep	9.3	B-061	3(3)	270	0.090-inch, max.
					3(4)	350	0.180-inch, max.
8	1B or 3	Weight Loss	---	B-005	3	350	1.25% max.
9	1B	Fluid Tightness	9.4	B-059	3	75	0.50-inch, max.
					3	180	0.50-inch, max.
10	3 or 4	Aluminum Overlap Shear Strength	9.6	MMM-A-132	5	75	2300 psi
					5	270	2300 psi
					5	350	1800 psi
11	3	Sandwich Flatwise Tension	9.2	B-057	6(1)	-65	2500 lb.
					6(1)	75	2500 lb.
					6(1)	270	2200 lb.
					6(1)	350	2000 lb.
12	3	Short Beam Sandwich Shear Strength	9.3	B-053	6(1)	-65	620 psi
					6(1)	75	620 psi
					6(1)	270	530 psi
					6(1)	350	480 psi

TABLE 2 (Concluded)

QUALIFICATION TEST REQUIREMENTS

No.	Adhesive Form	Property	Test Panel Para.	Test Method FPS-1028 (Unless Noted)	Number of Specimens	Test/Temp/ Condition(°F)	Minimum Required Average
13	4	Laminate-to-Aluminum Lap Shear	9.5	A-054	3	75	1600 psi
					3	270	750 psi
					3	350	540 psi
14	4	Short Beam Sandwich Shear	9.3	B-053	3	75	620 psi
					3	270	520 psi
					3	350	450 psi
15	N.A.	Salt Fog Exposure	---	ASTM B-117	---	---	---

NOTES:

(1) Test 1/2 of coupons without preconditioning and 1/2 of coupons after exposure of 300 hours at 270°F followed by 10 hours at 350°F. Acceptance tests are not preconditioned.
(2) Failure mode must be in core shear. Adhesive delamination is not acceptable.
(3) Load beam to core shear stress of 130 psi, hold 300 hours at 270°F.
(4) Load beam to core shear stress of 115 psi, hold 10 hours at 350°F.
(5) The coupon bondline shear stress ahll be constant during the loading period at each temperature as noted: 300 hours at 75°F under 2000 psi;
300 hours at 270°F under 1700 psi;
10 hours at 350°F under 1200 psi.
(6) Form 4 adhesive is cured at 270-280°F per Paragraph 9.19.
(7) Cure pressure is 25 ± 5 psi.
(8) Form 4 adhesive is intended for use per FPS-1067 or FPS-1081 or other applications where cure temperature is less than 300°F.

TABLE 3

ALUMINUM OVERLAP SHEAR STRENGTH,
QUALIFICATION TEST NO. 2 PER FMS-1013C

Form 1B[1] Adhesive, R.B. 398 N.A., Unconditioned

Test Condition	Shear Strength, psi[2]		Minimum Requirement, psi
	R-500 Primer	XEA-9289 Primer	
-65°F	4584	4020	2100
75°F	4585	3983	2300
270°F	4275	3484	2300
350°F	3604	2747	1800

Form 1B[1] Adhesive, R.B. 398 N.A., Conditioned 300 Hrs. @ 270°F Plus 10 Hrs. @ 350°F

Test Condition	Shear Strength, psi[2]		Minimum Requirement, psi
	R-500 Primer	XEA-9289 Primer	
-65°F	4154	3101	2100
75°F	3931	3510	2300
270°F	4217	3491	2300
350°F	4191	3218	1800

NOTES: (1) Form 1B adhesive cured 1 hour at 350°F and 45 psi.
(2) Average of five specimens.

TABLE 4

ALUMINUM OVERLAP SHEAR CREEP,
QUALIFICATION TEST NO. 3 PER FMS-1013C

Form 1B Adhesive, R.B. 398 N.A.

Test Condition	Creep, Inches[4] R-500 Primer	XEA-9289 Primer	Maximum Allowable, in.
75°F[1]	0.0038	0.0030	0.015
270°F[2]	0.0044[6]	0.0043[5]	0.015
350°F[3]			0.015

NOTES:
1. 300 hours at 72°F under 2000 psi.
2. 300 hours at 270°F under 1700 psi.
3. 10 hours at 350°F under 1200 psi.
4. Average of five specimens.
5. Average of three specimens. One specimen failed before 67 hrs. Another specimen failed during loading due to test oven overshooting while specimen was being stabilized at test temperature.
6. Average of two specimens.

TABLE 5

SANDWICH FLATWISE TENSION QUALIFICATION
TEST NO. 4 PER FMS-1013C

Form 1B Adhesive, R.B. 398 N.A., Unconditioned

Test Condition	Tensile Load, lbs. R-500 Primer	XEA-9289 Primer	Minimum[3] Requirement, lbs
-65°F	2233[1](7012)[2]	2224 (6983)	3375
75°F	2362 (7417)	2055 (6453)	3475
270°F	1850 (5809)	1724 (5413)	2900
350°F	1374 (4314)	1116 (3504)	2100

Form 1B Adhesive, R.B. 398 N.A., Conditioned 300 Hrs. @ 270°F Plus 10 Hrs. @ 350°F

Test Condition	Tensile Load, lbs. R-500 Primer	XEA-9289 Primer	Minimum[3] Requirement, lbs
-65°F	2253 (7074)	2139 (6716)	3375
75°F	2098 (6588)	2155 (6767)	3475
270°F	1661 (5216)	1351 (4242)	2900
350°F	980 (3080)	772 (2424)	2100

NOTES: (1) Failure load for 1-inch square specimen.
(2) Failure load extrapolated to a 2-inch diameter.
(3) Minimum requirement for 2-inch diameter specimen.

TABLE 6

SHORT BEAM SANDWICH SHEAR QUALIFICATION
TEST NO. 5 PER FMS-1013C

Form 1B Adhesive, RB 398 N.A.

Test Condition	Short Beam Sandwich Shear[1], lbs. R-500 Primer	XEA-9289 Primer	Minimum Requirement, lbs.
-65°F	3173	3178	2275
75°F	3220	3268	2275
270°F	3190	3175	2190
350°F	2647	2655	2000
400°F	1867	1925	1720

NOTE: 1. Average of three specimens.
All failures were core shear.

TABLE 7

LONG BEAM SANDWICH SHEAR
QUALIFICATION TEST NO. 6 PER FMS-1013C

Form 1B Adhesive, R.B. 398 N.A.

Test Condition	Long Beam Sandwich Shear[1] lbs. R-500 Primer	XEA-9289 Primer	Minimum Requirement, lbs.
-67°F	2854	2863[2]	2275
75°F	2717	2788[2]	2275
270°F	2363	2384	1800
350°F	2058	2046	1625
400°F	1788	1781[2]	1475

[1]Average of three specimens.
[2]Average of two specimens.

All failures were core shear.

TABLE 8

SANDWICH BEAM CREEP QUALIFICATION
TEST NO. 7 PER FMS-1013C

Form 1B Adhesive, RB398 N.A.

Test Condition	Sandwich Beam Creep[1] Inches		Maximum Requirements, In.
	R-500 Primer	XEA-9289 Primer	
270°F[2]	0.023	0.026	0.090
350°F[3]	0.029	0.028	0.180

[1]Average of three specimens.
[2]130 psi for 300 hrs. @ 270°F.
[3]115 psi for 10 hrs. @ 350°F.

TABLE 9

ADHESIVE WEIGHT LOSS
QUALIFICATION TEST NO. 8 PER FMS-1013C

Form 1B Adhesive, R.B. 398 N.A.

% Wt. Loss 1 hr. @ 350°F	Maximum Allowable Requirement (%)
0.45%	1.25%
0.46%	1.25%
0.43%	1.25%

Form 3 Adhesive, AF 130/2

% Wt. Loss 1 hr. @ 350°F	Maximum Allowable Requirement (%)
0.63%	1.25%
0.66%	1.25%
0.59%	1.25%

Test per FPS 1028 Method B-005.

TABLE 10

HONEYCOMB SANDWICH PANEL FLUID TIGHTNESS QUALIFICATION
TEST NO. 9 PER FMS-1013C

Form 1B Adhesive, R.B. 398 N.A.

Test Condition	Fuel Penetration,[1] inches R-500	XEA-9289	Maximum Requirement, lbs.
75°F	None	0.75	0.50
75°F	None	0.31	0.50
75°F	None	None	0.50
Avg.	0.0	0.35	
180°F	None	None	0.50
180°F	None	None	0.50
180°F	None	None	0.50
Avg.	0.0	0.0	
75°F	--	None[2]	0.50
75°F	--	None	0.50
75°F	--	None	0.50
75°F	--	None	0.50
75°F	--	None	0.50
75°F	--	None	0.50
Avg.	--	0.00	

[1]JP-4 per MIL-J-5624.
[2]Rerun fluid tightness samples.

TABLE 11

ALUMINUM OVERLAP SHEAR STRENGTH,
QUALIFICATION TEST NO. 10 PER FMS-1013C

Form 3[1] Adhesive, AF-130/2

Test Condition	Shear Strength, psi		Minimum Requirement, psi
	R-500 Primer	XEA-9289 Primer	
75°F	2424[3]	2490[3]	2300
270°F	2724	2736	2300
350°F	2823	2825	1800

Form 4[2] Adhesive, AF-130/2

Test Condition	Shear Strength, psi		Minimum Requirement, psi
	R-500 Primer	XEA-9289 Primer	
75°F	2638[3]	2515[3]	2300
270°F	3701	3205	2300
350°F	2650	2218	1800

NOTES: (1) Form 3 cured 1 hour at 350°F and 45 psi.

(2) Form 4 cured 3 hours at 275°F and 25 psi.

(3) Average of seven specimens, all others are average of five.

TABLE 12

SANDWICH FLATWISE TENSION
QUALIFICATION TEST NO. 11 PER FMS-1013C

Form 3 Adhesive, AF-130/2, Unconditioned

Test Condition	Tensile Load (lbs) R-500	XEA-9289	Minimum[3] Requirement (lbs)
-65°F	1480[1] (4649)[2]	1438[1] (4516)[2]	2500
75°F	1310 (4115)	1270 (3988)	2500
270°F	1192 (3742)	945 (2967)	2200
350°F	808 (2538)	793 (2489)	2000

Form 3 Adhesive, AF-130/2, Conditioned 300 hrs. @ 270°F + 10 hrs. @ 350°F

Test Condition	Tensile Load (lbs) R-500	XEA-9289	Minimum Requirement (lbs)
-65°F	1388[1] (4359)[2]	1278[1] (4014)[2]	2500
75°F	1695 (5322)	1448 (4548)	2500
270°F	1063 (3339)	1093 (3433)	2200
350°F	892 (2800)	861 (2704)	2000

NOTES:
1. Failure load for 1-inch square specimen.
2. Failure load extrapolated to a 2-inch diameter.
3. Minimum requirement for 2-inch diameter specimen.

TABLE 13

SHORT BEAM SANDWICH SHEAR
QUALIFICATION TEST NO. 12 PER FMS-1013C

Form 3[1] Adhesive, AF-130/2, Unconditioned

Test Condition	Short Beam Sandwich Shear[2](psi) R-500 Primer	XEA-9289 Primer	Minimum Requirement(psi)
-65°F	760	794	620
75°F	758	749	620
270°F	650	660	530
350°F	567	576	480

Form 3[1] Adhesive, AF-130/2, Conditioned 300 hrs. @ 270°F + 10 hrs. @ 350°F

Test Condition	Short Beam Sandwich Shear[2](psi) R-500 Primer	XEA-9289 Primer	Minimum Requirement(psi)
-65°F	820	814	620
75°F	787	802	620
270°F	676	689	530
350°F	643	644	480

NOTES: 1. Form 3 cured 1 hour @ 350°F and 45 psi.
2. Average of three specimens.
All failures were core shear.

TABLE 14

SHORT BEAM SANDWICH SHEAR
QUALIFICATION TEST NO. 14 PER FMS-1013C

Form 4[1] Adhesive, AF-130/2

Test Condition	Short Beam Sandwich Shear[2] (psi) R-500 Primer	XEA-9289 Primer	Minimum Requirement (psi)
75°F	786	754	620
270°F	706	674	520
350°F	585	563	450

NOTES: 1. Form 3 cured 3 hrs. @ 275°F and 25 psi.
2. Average of three specimens.

TABLE 15

ALUMINUM OVERLAP SHEAR STRENGTH AFTER SALT FOG AGING,
QUALIFICATION TEST NO. 15 PER FMS-1013C

Adhesive Form	Adhesive Primer	Tensile Lap Shear, psi Control	30 Day	90 Day
1B	R-500	4585	4723	4408
1B	XEA-9289	3983	2183	1938
1B	EC-3983	4947	3076	2553
3	R-500	2424	3257	3989
3	XEA-9289	2490	3281	3424
3	EC-3983	2562	4082	4326
4	R-500	2638	3645	4201
4	XEA-9289	2515	3549	4124
4	EC-3983	2898	4170	4659

NOTES: 1. Salt fog per ASTM B117, 95°F and 5% salt solution.
2. No minimum requirement in FMS-1013C, test is in addition to normal qualification.

TABLE 16

EA9394 ADHESIVE CHARACTERIZATION TEST MATRIX

Task Number	Variable	Condition	D1002 Lap Shear -65/RT/200	D3167 M-M Peel -65/RT/200	H/C Flat Tension/C297 -65/RT/200	D1781 H/C Peel -65/RT/200
1	Minimum Cure		0/0/30	0/0/30	---	---
2	Control	3 Batches	30/30/30	15/15/15	15/15/15	15/15/15
3	Humidity Exposure	3 Batches	15/15/15	15/15/15	15/15/15	0/15/0
4	Primer Thickness	1 Thickness	5/5/5	5/5/5	---	---
5	Adhesive Thickness	2 Thicknesses	10/10/10	10/10/10	---	---
6	Overlap Length	3 Overlaps	0/15/0	---	---	---
7	Adherends	3 Materials	30/30/30	---	---	---
8	Effect of 350°F Cure	2 Exposure Times	---	0/10/10	---	---
9	T_g	3 Batches Dry & Wet	---	---	---	---
10	Non-primed Surface	2 Adherend Materials Several Surf. Cond.	---	---	---	---
11	Storage & Elev. Temp Aging	3 Temps. Various Times	TBD	---	---	---
12	Pot Life	2 Temps. 2 Times	0/20/20	20/20/20	---	---

TABLE 16(Continued)

EA93[illegible]4 ADHESIVE CHARACTERIZATION TEST MATRIX

Task Number	Variable	Condition	Lap Shear -65/RT/200	M-M Peel -65/RT/200	H/C Flat Tension -65/RT/200	H/C Peel -65/RT/200
13	Creep Durability	120°F/100% RH D2919	---	---	---	---
14	Thermal Pulse	Dry and Wet Painted	0/10/0	0/10/0	---	---
15	Fatigue		---	---	---	---

TABLE 17

ISOTHERMAL DSC TO DETERMINE MINIMUM CURE FOR EA9394 ADHESIVE, HEAT-UP RATE 1°C/MIN.

Iso-Thermal Temperature, (°C)	(°F)	Degree of Cure (%)	Time at Temperature (min.)	Heat of Reaction (J/g)
52	(125)	100	95	157
		90	45	140
		80	35	124
66	(150)	100	61	273(133)[2]
		90	15	245(138)
		80	9	218(122)
79	(175)	100	77	239
		90	9	215
		80	1	191
93	(200)	100	40	363
		90	0[1]	326
		80	0[1]	290

NOTES: (1) Sample reached designated percent cure before iso-thermal temperature.

(2) Sample rerun at 66°C (150°F).

TABLE 18

CURE STATE OF EA9394 ADHESIVE AT SPECIFIC TEMPERATURES ARE DETERMINED BY ISOTHERMAL DSC

Type Run	Heating Rate (°C/min.)	Maximum Temperature (°C)	(°F)	Time at Temperature (min.)	Heat of Reaction (J/gm)	% of Cure[1]	Residual Cure (J/gm)	Total Heat of Reaction (J/gm)
Dynamic	1	229	(444)	---	385	---	---	385
Isothermal	1	52	(125)	95	157	41	59	216
Isothermal	1	66	(150)	61(60)[2]	273(153)[2]	71(40)[2]	106(86)[2]	379(239)[2]
Isothermal	1	80	(175)	77	239	62	72	311
Isothermal	1	93	(200)	40	363	---	92	455

NOTES: 1. Percent of cure which can be achieved at 200°F after 1 hour.

2. Sample rerun at 66°C (150°F).

TABLE 19

TENSILE LAP SHEAR STRENGTH VS.
CURE TEMPERATURE AND TIME

Cure Condition	Lap Shear (psi)		Peel (in·lbs/in)	
	R.T.(1)	200°F(2)	R.T.	200°F
1 hr. @ 200°F	4026	3192	13.3	
1 hr. @ 125°F	3326	2524		
1 hr. @ 150°F	3469	2619		
1 hr. @ 175°F	3291	2319		
2 hrs. @ 125°F	3014	2239		
2 hrs. @ 150°F	4378	3433	18.6	
2 hrs. @ 175°F	4370	3389	18.0	
24 hrs. @ R.T.	3412	2974		
72 hrs. @ R.T.	2720	1992		
168 hrs. @ R.T.	2795	2325		

NOTES: 1. Average of four specimens.
2. Average of three specimens.

TABLE 20

EFFECTS OF THE TYPE BONDLINE CONTROL AND PRESSURE APPLICATION, TENSILE LAP SHEAR, PSI

Type Cure and Pressure Application	Type Bondline Control		
	Beads	Scrim	None
Full vacuum 200°F for 1 hour Heat-up-rate, 2°F/min.	3009 (0.006)[1]	3134 (0.007)	3542 (0.003)
Dead weight 10 ± 2 PSI R.T. for 7 days	3993 (0.007)	3939 (0.010)	4211 (0.004)
Partial Vacuum (12-15, in. Hg) 200°F for 1 Hour Heat-up-rate, 2°F/min.	4540 (0.006)	4194 (0.007)	
Press Cure 15 PSI 200°F for 1 Hour 4.5°F/min.	2848 (0.005)	3482 (0.005)	

NOTE: 1. Glueline thickness.

TABLE 21

EFFECTS OF TIME DELAY AT WHICH THE TEMPERATURE AND/OR FULL VACUUM PRESSURE IS APPLIED TO EA9394 ADHESIVE BONDED JOINTS

Time Delay, min.		Glueline Thickness (in.)	Lap Shear Strength (psi)
Pressure	Temperature		
0	0	0.007	3134
0	45	0.007	3374
0	90	0.007	3138
15	15	0.006	4435
30	30	0.006	4273
45	45	0.007	4241
90	90	0.007	4060

TABLE 22

EFFECTS OF VACUUM PRESSURE UPON GLUELINE THICKNESS AND LAP SHEAR STRENGTHS FOR EA93934 ADHESIVE

Vacuum (in. Hg)	Glueline Thickness (in.)	Lap Shear Strength (psi)
27	0.007	3134
20-22	0.007	4183
12-15	0.007	4194
6-8	0.007	4244

TABLE 23

CONTROL DATA, TENSILE LAP SHEAR AND FLOATING ROLLER PEEL FOR THREE PRODUCTION BATCHES OF EA9394 ADHESIVE, AVERAGE AND STANDARD DEVIATION

Adhesive Batch Number		Tensile Lap Shear Strength (psi)[1] -65°F	R.T.	200°F	Floating Roller Peel[2] (lbs/in) -65°F	R.T.	200°F
8204	Avg.	3535	4026	3192	12.2	13.3	12.4
	S.D.	218	341	473	2.8	1.7	1.5
8221	Avg.	3864	4573	3424	11.2	11.5	12.1
	S.D.	464	362	217	1.1	1.7	1.0
8359	Avg.	3621	4172	3419	10.0	12.2	12.2
	S.D.	334	432	208	1.3	1.2	2.6

NOTES: 1. Average of 10 specimens.

2. Average of 5 specimens.

TABLE 24

SUMMARY OF TENSILE LAP SHEAR RESULTS SINCE MODIFICATION OF VACUUM APPLICATION SYSTEM

Fabrication Procedure	Bond Line Appearance	Bond Line Thickness	Lap Shear Strength, psi
"Standard Cure", 45-min. hold after mixing adhesive(1)	Foamy	0.012	3474
Same as above	Foamy	0.011	2900
"Standard Cure", 25-min. hold after mixing adhesive	Foamy	0.013	2777
Same as above, except fresh adhesive	Foamy	0.007	3253
R.T. cure, 10 psi dead wt. load	Good	0.009	3398
"Standard Cure", 45-min. hold after placed in oven	Foamy	0.012	2928
"Standard Cure", 90-min. hold after placed in oven	Foamy	0.012	2988
"Standard Cure", no hold, aged adhesive	Foamy	0.007	3349
"Standard Cure", 90-min. hold after placed in oven, attention to bond line	Foamy	0.007	3629
"Standard Cure", 45-min. hold after placed in oven, 20 to 22 in/Hg vac.	Slight foam	0.007	3838
"Standard Cure" 45-min. hold after placed in oven, 16 in./Hg vac	Very Good	0.007	3875

NOTE: (1) "Standard Cure" = 1 hour at 200°F under vacuum pressure.

TABLE 25

TENSILE LAP SHEAR RESULTS FOR FRESH
VS. OLD EA9394 ADHESIVE

Lot No.	Date Fabricated	Cure Cycle		Processing Delay	Tensile Lap Shear, PSI	
		Time & Temp.	Pressure, in. Hg		R.T.	200°F
8204[1]	1-89	1 hr. @ 200°F	28[1]	None	3134	--
8204	1-89	1 hr. @ 200°F	13	None	4194	--
8204	1-89	1 hr. @ 200°F	28	45 mins.	4026	3192
8204	3-89	2 hrs. @ 175°F	28	45 mins.	4370	3389
8204	6-89	1 hr. @ 200°F	28[2]	45 mins.	2949	1670
8204	6-89	1 hr. @ 200°F	11	45 mins.	3695	2368
9114[2]	7-89	1 hr. @ 200°F	28	None	3473	2157
9114	7-89	1 hr. @ 200°F	10	None	3662	2245
9114	7-89	1 hr. @ 200°F	28	45 mins.	3457	2152
9114	7-89	1 hr. @ 200°F	10	45 mins.	3916	2892
9114	7-89	2 hrs. @ 175°F	28	45 mins.	3654	2437
9114	7-89	2 hrs. @ 175°F	10	45 mins.	4280	3033

[1]Vacuum pressures recorded before 6-89 may have been considerably lower than that indicated.

[2]Vacuum pressures recorded 6-89 and after are accurate.

[3]Lot No. 8204 was manufactured 6-23-88.

[4]Lot No. 9114 was manufactured 4-24-89.

TABLE 26

TENSILE LAP SHEAR STRENGTHS FOR EA9394
ADHESIVE VS. PROCESSING TECHNIQUE

Pressure Application	Bondline Pressure(psi)	Lot 8359 Shear (psi) R.T.	200°F
Dead Weight	5	4503	3460
Dead Weight	10	4127	3277
Dead Weight	15	4552	3396
Single Bag	5	4440	3084
Single Bag	10	3875	2325
Single Bag	15	3789	2566
Envelope Bag	5	4230	3251
Envelope Bag	10	4056	3276
Envelope Bag	15	4247	3450

TABLE 27

TENSILE LAP SHEAR, PSI, DRY VS. WET

	-65°F[1]		R.T.		200°F[1]	
Batch No.	Dry	Wet[2]	Dry	Wet[2]	Dry	Wet[2]
8204	3535	4134	4026	3658	3192	2873
8221	3864	4334	4573	4090	3424	2982
8359	3621	4283[3]	4172	3872	3419	3072

NOTES: 1. Dry specimens soaked at temperature for 10 mins.
Wet specimens soaked at temperature for 4 mins.

2. 30 days at 140°F and 95-100% R.H.

3. One wet specimen soaked for 10 mins. @ -65°F,
lap shear = 4603 psi.

TABLE 28

FLOATING ROLLER PEEL, lbs/in, DRY VS. WET

	-65°F[1]		R.T.		200°F[1]	
Batch No.	Dry	Wet[2]	Dry	Wet[2]	Dry	Wet[2]
8204	12.2	11.5	13.3	11.8	12.4	8.8
8221	11.2	10.9	11.5	12.0	12.1	10.3
8359	10.0	10.2	12.2	11.3	12.2	10.3

NOTES: 1. Dry specimens soaked at temperature for 10 mins.
Wet specimens soaked at temperature for 4 mins.

2. 30 days at 140°F and 95-100% R.H.

TABLE 29

STORAGE AND ELEVATED TEMPERATURE AGING
EFFECT ON EA9394 ADHESIVE

Storage Temp.	Storage Time	HPLC		IR		Viscosity, poise[1]		DSC	RSA	R.T. Lap Shear[2]	200°F Lap Shear[3]
		Part A	Part B	Part A	Part B	Part A	Part B				
Initial	0	✓[4]	✓	---	---	10,820	540	✓	✓	3051	2378
72°F	1 mo.	✓		✓	✓	12,100	650	✓	✓	3217	2649
	6 mos.			✓	✓	13,340	566	✓	✓	4077	3580
	9 mos.			✓	✓	11,820	684	✓		3101	1930
	12 mos.			✓	✓	11,940	710	✓		3626	2639
	18 mos.										
100°F	1 mo.	✓		✓	✓	11,100	634	✓	✓	3175	2746
	6 mos.			✓	✓	11,300	768	✓	✓	3871	3307
	9 mos.			✓	✓	12,140	702	✓		2932	1919
	12 mos.			✓	✓	12,900	730	✓		3528	2628
	18 mos.										
120°F	1 mo.	✓		✓	✓	13,260	663	✓	✓	2956	2412
	6 mos.			✓	✓	12,940	818	✓	✓	2686	3656
	9 mos.			✓	✓	13,440	880	✓		3148	1971
	12 mos.			✓	✓	15,160	868	✓		3343	2472
	18 mos.										

NOTES:
1. Brookfield Viscometer Model RVF.
2. Average of three tests.
3. Average of four tests.
4. ✓ - completed.

TABLE 30

TENSILE LAP SHEAR STRENGTH, psi, OF
EA9394 ADHESIVE AFTER 12-MONTH STORAGE

Storage Condition	Envelope Bag		Single Bag	
	R.T.	200°F	R.T.	200°F
12 mos. @ R.T.	4237	3563	3626	2639
12 mos. @ 100°F	4254	3511	3528	2628
12 mos. @ 120°F	4228	3312	3343	2472

NOTE: Adhesive was manufactured three months before aging had begun.

TABLE 31

WEDGE-CRACK PROPAGATION BEHAVIOR OF
BONDED ALUMINUM PANELS PREPARED WITH VARIOUS
WINDECKER/PRIMER OR PAA/PRIMER FORMULATIONS (WT. RATIOS)

PAA/Primer or Windecker/ Primer Formulations (% by wt.)	Initial Crack Length inch (mm)	Crack Length 1 Hour inch (mm)	Crack Growth 1 Hour inch (mm)	Crack Length 24 Hours inch (mm)	Crack Growth 24 Hours inch (mm)	Crack Length 1 Week inch (mm)	Crack Growth 1 Week inch (mm)	Remarks
Phosphoric acid anodize/BR-127 primed (PAA/ BR-127)	1.19 (30)	1.19 (30)	--- ---	1.24 (32)	0.05 (1)	1.29 (33)	0.10 (3)	Controls
Phosphoric acid anodized with 20/80/1 as primer	1.27 (32)	1.29 (33)	0.02 (0.5)	1.32 (34)	0.05 (1)	1.33 (34)	0.06 (2)	20% Adhesive; 80% Acetone; 1% Z6020 Silane
Phosphoric acid anodized with 50/50/1 as primer	1.26 (32)	1.27 (32)	0.01 (0.3)	1.31 (33)	0.05 (1)	1.35 (34)	0.09 (2)	50% Adhesive; 50% Acetone; 1% Z6020 Silane
Modified quick windecker 20/80/1	1.28 (33)	1.40 (36)	0.12 (3)	2.26 (57)	0.98 (25)	3.25 (83)	1.97 (50)	20% Adhesive; 80% Acetone; 1% Z6020 Silane

TABLE 32

WEDGE-CRACK PROPAGATION BEHAVIOR OF BONDED 2024-T3 BARE ALUMINUM PANELS PREPARED WITH VARIOUS COMBINATIONS OF SURFACE PREPARATIONS AND PRIMER

Panel Number	Surface Preparation Procedure	Primer	Adhesive	Hydrolysis Time Allowed	Initial Crack Length (inch)	One Hour Length (inch)	One Hour Growth (inch)	Four Hour Length (inch)	Four Hour Growth (inch)	24 Hour Length (inch)	24 Hour Growth (inch)	48 Hour Length (inch)	48 Hour Growth (inch)	72 Hour Length (inch)	72 Hour Growth (inch)	120 Hour Length (inch)	120 Hour Growth (inch)	7 Day Length (inch)	7 Day Growth (inch)	28 Day Length (inch)	28 Day Growth (inch)	Failure Mode (% Coh)
1	PAA[1]	BR127	FM-123-2	N.A.	1.29	1.32	0.03	---	---	1.35	0.06	1.38	0.09	---	---	1.39	0.10	1.40	0.11	1.40	0.11	100
2	PAA[1]	BR127	EA9628	N.A.	1.42	1.46	0.04	1.50	0.08	---	---	---	---	---	---	---	---	1.58	0.16	1.58	0.16	100
3	PAA[1]	3 coats 1% E8385 Soln.[5]	EA9628	60 min.	1.44	1.55	0.11	1.58	0.14	1.69	0.25	---	---	1.88	0.44	---	---	1.97	0.53	2.20	0.76	100
4	Dry SBA[2]	BR127	EA9628	N.A.	1.85	3.65	1.80	3.69	1.84	3.73	1.88	---	---	3.73	1.88	---	---	3.77	1.92	3.77	1.92	0
5	Dry SBA[2]	3 coats 1% E8385 Soln.[5]	EA9628	20 min.	1.59	1.80	0.21	2.03	0.44	2.53	0.94	---	---	2.60	1.01	---	---	2.77	1.18	3.00	1.41	0
6	Dry SBA[2]	3 coats 1% E8385 Soln.[5]	EA9628	60 min.	1.62	1.73	0.11	1.93	0.31	2.38	0.76	---	---	2.54	0.92	---	---	2.72	1.10	3.09	1.47	0
7	Dry SBA[2]	1 coat 3% E8385 Soln.[5]	EA9628	60 min.	1.59	1.72	0.13	1.79	0.20	1.99	0.40	---	---	2.07	0.48	---	---	2.22	0.63	2.57	0.98	0
8	Wet SBA-1[3]	3 coats 1% E8385 Soln.[5]	EA9628	20 min.	1.46	1.56	0.10	1.62	0.16	2.37	0.91	---	---	2.43	0.97	---	---	2.48	1.02	2.78	1.32	0
9	Wet SBA-1[3]	3 coats 1% E8385 Soln.[5]	EA9628	60 min.	1.50	1.56	0.06	1.61	0.11	2.31	0.81	---	---	2.50	1.00	---	---	2.62	1.12	2.94	1.44	0
10	Wet SBA-2[4]	1 coat 25% Z1-6100 Soln.[6]	EA9628	N.A.	1.98	2.15	0.17	---	---	---	---	---	---	2.50	0.52	---	---	2.76	0.78	2.87	0.89	0

[1]Modified Phosphoric Acid Anodization - alkaline wash and sulfuric acid-dichromate deoxidizing steps replaced by a dry Scotchbrite abrasion process and water rinse.

[2]This surface preparation consisted of dry abrasion with a Scotchbrite pad and a water rinse.

[3]This surface preparation consisted of a wet Scotchbrite abrasion process in which a 1% solution of E8385 in toluene was used as the abrasion and rinse solution.

[4]This surface preparation consisted of a wet Scotchbrite abrasion process in which a 1% solution of Z6020 in water was used as the abrasion and rinse solution. This solution was allowed to stand 1 hour after mixing before use.

[5]The primer solution was 1% or 3% E8385 (by wt.) in toluene, as noted. After application, the toluene rapidly flashed off and the primer was allowed to stand at ambient conditions for 20 or 60 minutes to permit hydrolysis to occur. The hydrolysis reaction depended on the presence of water left over from the surface preparation and/or atmospheric moisture.

[6]The primer solution was 25% Z1-6100 (by wt.) in isopropyl alcohol.

[7]The indicated failure mode was for the propagation portion of the crack growth. In all cases the initial crack length exhibited 100% cohesive failure.

TABLE 33

A357 CHEMICAL COMPOSITION

Elements	Percent, Minimum	Percent, Maximum
Copper	---	0.20
Silicon	6.5	7.5
Iron	---	0.10
Manganese	---	0.10
Zinc	---	0.10
Magnesium	0.55	0.65
Titanium	0.10	0.20
Beryllium	0.04	0.07
Others, each	---	0.05
Others, total	---	0.15
Aluminum	Remainder	

TABLE 34

A357 HEAT TREATMENT

Solution Heat Treatment	Quench Delay	Quenchant	Natural Aging	Precipitation Heat Treatment (Aging)
1010°F ± 10°F for 16 hrs. min. [1]	8 sec. max.	170°F ± 30°F water	Room temp. for 16-24 hrs.	325°F ± 10°F for 8 hrs ± 1 hr

[1] For castings with 1-inch maximum thickness. Add 2 hours soak for each additional 1/2-inch thickness.

TABLE 35

LAP SHEAR TEST MATRIX

250°F Adhesive System

- R.T., dry
- 180°F, dry
- R.T., after 2 wks. @ 120°F, 100% R.H.
- 180°F, after 2 wks. @ 120°F, 100% R.H.

350°F Adhesive System

- R.T., dry
- 300°F, dry
- 350°F, dry
- R.T., after 2 wks. @ 140°F, 100% R.H.
- 300°F, after 2 wks. @ 140°F, 100% R.H.
- 350°F, after 2 wks. @ 140°F, 100% R.H.

Replications, 5 at each data point with both types of adherends.

TABLE 36

WEDGE CRACK TEST MATRIX

250°F Adhesive System

- Salt spray aging, 3 mos. @ 95°F per ASTM B117
- Humid aging, 3 mos. @ 120°F, 100% R.H.

350°F Adhesive System

- Salt spray aging, 3 mos. @ 95°F per ASTM B117
- Humid aging, 3 mos. @ 160°F, 100% R.H.

Reading of crack length was taken at following times: at initial penetration, 1 hr., 4 hrs., 8 hrs., 24 hrs., 48 hrs., 7 days, 14 days, 1 month, 2 months, 3 months.

Replications, 5 at each data point with both types of adherends.

TABLE 37

TENSILE LAP SHEAR DATA,
A357 CAST ALUMINUM vs. 2024-T3 SHEET ALUMINUM

Adhesive System	Test Condition	Aging Condition	Cast Aluminum Lap Shear Strength (psi)	2024-T3 Aluminum Lap Shear Strength (psi)
250°F (EA 9628H)	R.T.	Dry	5690	6510
		Wet	5430	6390
	180°F	Dry	4090	4590
		Wet	3350	4240
350°F (FM-300)	R.T.	Dry	4300	4410
		Wet	4730	4800
	300°F	Dry	1660	2570
		Wet	1780	2440
	350°F	Dry	570	460
		Wet	320	430

TABLE 38

WEDGE CRACK GROWTH DATA,
250°F CURING ADHESIVE SYSTEM,
A357 CAST ALUMINUM vs. 2024-T3 SHEET ALUMINUM

Time	Salt Spray Aging 5% Salt Fog & 95°F		Humid Aging, 120°F and 95-100% R.H.	
	Cast Aluminum EA 9628H	2024-T3 Aluminum EA 9628NW	Cast Aluminum EA 9628H	2024-T3 Aluminum EA 9628NW
Initial	1.3927	1.2957	1.3368	1.3058
1 Hr.	0.0066	0.0288	0.0200	0.0598
4 Hrs.	0.0131	0.0492	0.0200	0.0864
8 Hrs.	0.0195	0.0544	0.0200	0.0916
24 Hrs.	0.0289	0.0630	0.0287	0.0980
48 Hrs.	0.0289	0.0830	0.0287	0.1006
7 Days	0.0456	0.1330	0.0405	0.1227
14 Days	0.0609	0.1719	0.0580	0.1518
1 Month	0.0609	0.1719	0.0628	0.2387
2 Months	0.1390	0.1836	0.1623	0.2899
3 Months	0.1390	0.1993	0.1623	0.3056

TABLE 39

WEDGE CRACK GROWTH DATA,
350°F CURING ADHESIVE SYSTEM (FM-300),
A357 CAST ALUMINUM vs. 2024-T3 SHEET ALUMINUM

Time	Salt Spray Aging		Humid Aging	
	Cast Aluminum	2024-T3 Aluminum	Cast Aluminum	2024-T3 Aluminum
Initial	1.6430	1.8282	1.6033	1.8779
1 Hr.	0.0436	0.0168	0.0714	0.0323
4 Hrs.	0.0588	0.0444	0.0748	0.0345
8 Hrs.	0.0691	0.0444	0.0748	0.0388
24 Hrs.	0.0757	0.0632	0.0842	0.0472
48 Hrs.	0.0957	0.0688	0.0842	0.0472
7 Days	0.1045	0.0688	0.0896	0.0548
14 Days	0.1045	0.0779	0.0993	0.0675
1 Month	0.1045	0.0779	0.1081	0.0675
2 Months	0.1045	0.1039	0.1152	0.0765
3 Months	0.1157	0.1039	0.1298	0.0765

TABLE 40

LAP SHEAR STRENGTH OF HIGH TEMPERATURE
ADHESIVES ON TITANIUM ADHERENDS

Test Condition	NR-150 (psi)	NR-150 [MPa]	IP-600 (psi)	IP-600 [MPa]	FA-7001 (psi)	FA-7001 [MPa]				
0.17 hr. @ -65°F (-54°C)										
72°F (22°C)	2275	[15.7]	2425	[16.7]	1949	[13.4]				
0.17 hr. @ 600°F (316°C)	1374	[9.5]	1479	[10.2]	961	[6.6]				
0.5 hr. @ 600°F (316°C)	1385	[9.5]	1386	[9.5]	1041	[7.2]				
24 hrs. @ 600°F (316°C)	1460	[10.1]	1343	[9.3]	1560	[10.8]				
0.1 hr. @ 800°F (427°C)	293	[2.0]	638	[4.4]						
0.5 hr. @ 800°F (427°C)	371	[2.6]	567	[3.9]						
0.1 hr. @ 1000°F (538°C)	268	[1.9]	529	[3.7]						

TABLE 41

LAP SHEAR STRENGTH OF HIGH TEMPERATURE ADHESIVES ON TITANIUM ADHERENDS

Test Condition	NR-150		IP-600		FA-7001			
	(psi)	[MPa]	(psi)	[MPa]	(psi)	[MPa]	(psi)	[MPa]
Wet[1]								
72°F (22°C)	1510	(10.4)						
0.05 hr. @ 600°F (316°C)			1340	(9.25)				
Dry								
72°F (22°C)	2275	(15.7)	2425	(16.7)	2042	(14.1)		
0.17 hr. @ 600°F (316°C)	1374	(9.5)	1479	(10.2)	1357	(9.36)		

[1]After 2 Weeks @ 160°F (71°C) and 95-100% R.H.

TABLE 42

EA9396 COMPOSITE CHARACTERIZATION TEST MATRIX
7781 GLASS AND W133 GRAPHITE COMPOSITES

Task Number	Property	No. of Replicates at Each Condition and Temperature: Dry -65/RT/200	Wet -65/RT/200
1	Degree of Cure (Four-Point Shear)	---	---
2	Longitudinal (Warp) Tension D3039 F^{tu}, ε^{ult}, E^{t}, ν_{12}	0/15/0	0/15/0
3	Transverse (Fill) Tension D3039	15/15/15	15/15/15
4	Longitudinal (Warp) Compression F^{cu}, ε^{ult}, E^{c} D3410	0/15/0	0/15/0
5	Transverse (Fill) Compression F^{cu}, ε^{ult}, E^{c} D3410	15/15/15	15/15/15
6	45° Tension/Inplane Shear F^{ipsu}, E^{t}, G D3518	15/15/15	15/15/15
7	Interlaminar Shear (Four-Point)	0/15/0	0/15/15
8	350°F Cure Effect Interlaminar (Four-Point) Shear	0/6/0	0/0/6
9	Effect of Co-cure on Honeycomb Sandwich Beam Compression Transverse (Fill)	0/3/3	0/3/0
10	Bearing Strength MIL-HDBK-17	0/3/0	0/3/0
11	T_g	9	9
12	Effect of Resin Content D3410		
	Four-Point Shear (Warp)	0/15/0	---
	Transverse (Fill) Compression	0/15/0	---
13	Effect of Resin Storage and Elevated Temperature		
	Four-Point Shear (Warp)	0/TBD/0	---
	Viscosity	---	---
	Heat of Cure	---	---
	Viscosity Cure Profile	---	---
	FTIR Spectra	---	---
	HPLC Spectra	---	---

TABLE 42 (Concluded)

EA9396 COMPOSITE CHARACTERIZATION TEST MATRIX
7781 GLASS AND W133 GRAPHITE COMPOSITES

Task Number	Property	No. of Replicates at Each Condition and Temperature: Dry -65/RT/200	Wet -65/RT/200
14	Pot Life Four-Point Shear (Warp)	0/TBD/0	---
15	Thermal Flash Effects D3410 Transverse (Fill) Compression	0/5/5	5/5/5
16	Effect of Cure Time and Temperature Four-Point Shear (Warp)	0/5/0	0/5/0
17	Effect of Vacuum Level Four-Point Shear (Warp)	0/5/0	0/5/0
18	Effect of Processing Layup Variables Perforated Bagging Film Double Bagging Alternate Impregnation Methods Deaeration Agent	0/5/0	0/5/0
19	Effect of A1100 Sizing Tension (Warp); F^{tu}, E^{t}, ε^{ult} Inplane Shear; F^{ips}, E^{t}, G Four-Point Shear; F^{ils}	0/5/0 0/5/0 0/5/0	0/5/5 0/5/5 0/5/5
20	Alternate Bleeder Materials	---	---

NOTES:

1. Tasks 2, 3, 4, 5, 6, 7 and 11 each involved the use of three separate resin lots. One-third of the indicated number of replicate tests for each of these tasks represent each resin lot.

2. Tasks 1, 8, 9, 10, 12, 13, 14, 15, 16, 17, 18, and 19 involved only one resin lot.

TABLE 43

RESULTS OF DSC CURE STUDIES ON EA9396 A/B RESIN

Type DSC Test	Heating Rate (°C/min)	Hold/Max. Temperature (°C)	(°F)	Time Needed to Complete Cure at Hold Temperature (min.)	Maximum Exotherm (J/gm)	Cure State (% of 535 J/gm)	Residual Exotherm (J/gm)	Total Exotherm (J/gm)
Dynamic	10	300	572	---	535	100	---	535
	1	225	437	---	574	107	---	574
Isothermal	---	22	72	(given 7 days)	---	68 (est)	170	535(est)[3]
	2	66	150	~ 75	450	83	94	544
	2	82	180	~ 40	479	89	57	536
	2	82	180	~ 41	519	90	60[1]	579[2]
	2	93	200	~ 30	486	91	46	532
	2	107	225	~ 10	496	95	27	522

NOTES:
1. Time after reaching hold temperature.
2. After 82°F isothermal cure, sample held at 22°C for 7 days before residual heat of cure was determined in a dynamic DSC test.
3. Specimen was cured at 22°C for 7 days before DSC test for residual exotherm was conducted.

TABLE 44

EFFECT OF CURE TEMPERATURE ON INTERLAMINAR SHEAR STRENGTH OF W-133 GRAPHITE-REINFORCED EA9396 LAMINATES

Cure Condition(1)	Interlaminar Shear Strength (psi)			
	72°F, Dry	200°F, Dry	72°F, Wet	200°F, Wet
45 mins. at 225°F	6120		4540(4)	
45 mins. at 200°F(2)	5650-5780(typ)		4600-4750(typ)	2000-2700(typ)
30 mins. at 200°F	6220	4300	4430(3)	2270(3)
45 mins. at 175°F	5640		4030(4)	
4 hrs. at 72°F under vacuum pressure, plus additional 3 mos. at 72°F or wet aging	5010		3410(3)	

NOTES:

(1) All cures were under nominally full vacuum pressure (~23-25 in. Hg).

(2) This represents the "standard" cure schedule, used throughout this program.

(3) These specimens were wet-aged at 140°F, 95-100% R.H. to saturation.

(4) These specimens were wet-aged by water immersion at 140°F until saturated.

TABLE 45

LONGITUDINAL (WARP) TENSILE PROPERTIES OF GLASS-REINFORCED E-7781/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch No.	Ultimate Strength (psi)	Tensile Modulus (10^6 psi)	Ultimate Strain (10^{-6}in/in)	Poisson's Ratio
72°F, DRY	8235 - Avg.	51,740	3.600	17,480	0.121
	Std.Dev.	2,410	0.084	970	---
	8264 - Avg.	53,450	3.680	18,510	0.110
	Std.Dev.	3,410	0.088	2,040	---
	8284 - Avg.	50,090	3.583	17,240	0.114
	Std.Dev.	750	0.081	560	---
72°F, WET	8235 - Avg.	16,400	3.182	5,283	0.076
	Std.Dev.	810	0.095	438	---
	8264 - Avg.	15,430	3.273	4,795	0.092
	Std.Dev.	1,330	0.067	484	---
	8284 - Avg.	17,330	3.437	5,213	0.092
	Std.Dev.	970	0.061	328	---

NOTES: 1. All avg. values represent five specimens except Poisson's Ratio, which only represents two specimens.

2. WET = wet aged at 140°F/100% R.H. until saturated.

TABLE 46

TRANSVERSE (FILL) TENSILE PROPERTIES OF GLASS-REINFORCED E-7781/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch No.	Ultimate Strength (psi)	Tensile Modulus (10^6psi)	Ultimate Strain (10^{-6}in/in)	Poisson's Ratio
-65°F, Dry	8235 - Avg.	76,350	4.243	23,110+	0.153
	Std.Dev.	2,890	0.043	3,300	---
	8264 - Avg.	69,150	4.205	18,160+	0.155
	Std.Dev.	10,040	0.263	4,980	---
	8284 - Avg.	70,321	4.113	20,420+	0.155
	Std.Dev.	1,100	0.120	6,510	---
72°F, Dry	8235 - Avg.	53,340	3.922	17,050	0.124
	Std.Dev.	4,750	0.193	1,490	---
	8264 - Avg.	53,400	3.542	18,450	0.128
	Std.Dev.	2,430	0.097	1,320	---
	8284 - Avg.	56,080	3.545	20,770+	0.130
	Std.Dev.	2,060	0.102	2,950	---
200°F, Dry	8235 - Avg.	46,550	3.547	15,200	0.116
	Std.Dev.	2,670	0.099	820	---
	8264 - Avg.	45,280	3.515	14,900	0.095
	Std.Dev.	1,830	0.099	550	---
	8284 - Avg.	43,790	3.517	13,010+	0.094
	Std.Dev.	4,230	0.116	2,270	---
-65°F, Wet	8235 - Avg.	19,530	3.880	5,550	0.143
	Std.Dev.	2,780	0.078	1,040	---
	8264 - Avg.	22,720	3.764	6,830	0.117
	Std.Dev.	2,230	0.206	460	---
	8284 - Avg.	21,390	3.780	6,350	0.145
	Std.Dev.	2,130	0.095	660	---
72°F, Wet	8235 - Avg.	18,750	3.262	6,060	0.097
	Std.Dev.	1,060	0.090	320	---
	8264 - Avg.	16,350	3.965	4,410	0.016
	Std.Dev.	640	1.051	1,220	---
	8284 - Avg.	17,530	3.192	5,770	0.086
	Std.Dev.	1,580	0.033	480	---
200°F, Wet	8235 - Avg.	13,610	2.872	4,630	0.048
	Std.Dev.	580	0.055	150	---
	8264 - Avg.	13,520	3.237	4,300	0.101
	Std.Dev.	1,320	0.411	710	---
	8284 - Avg.	13,500	2.912	4,490	0.087
	Std.Dev.	1,040	0.385	430	---

NOTES: 1. All avg. values represent 5 specimens except Poisson's ratio, only represents 2 specimens.
2. WET = wet aged at 140°F, 100% R.H. until saturated.

TABLE 47

LONGITUDINAL (WARP) TENSILE PROPERTIES OF GRAPHITE - REINFORCED T300-W133/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch No.	Ultimate Strength (psi)	Tensile Modulus (10^6psi)	Ultimate Strain (10^{-6}in/in)	Poisson's Ratio
72°F, Dry	8235 - Avg.[1]	82,300	8.380	8210	0.053
	Std.Dev.	4,710	0.377	570	---
	8264 - Avg.	81,370	8.184	7510	0.053
	Std.Dev.	4,310	0.346	840	---
	8284 - Avg.	78,230	8.562	7060	0.073
	Std.Dev.	6,400	0.410	1020	---
72°F, Wet[2]	8235 - Avg.	86,670	9.050	9620	0.029
	Std.Dev.	5,830	0.212	600	---
	8264 - Avg.	81,560	8.642	9280	0.041
	Std.Dev.	4,960	0.235	470	---
	8484 - Avg.	86,480	8.780	9830	0.041
	Std.Dev.	3,370	0.282	370	---

NOTES: (1) All avg. values represent five specimens except Poisson's ratio, which only represents two specimens.

(2) WET = Wet aged at 140°F, 100% relative humidity until saturated.

TABLE 48

TRANSVERSE (FILL) TENSILE PROPERTIES OF GRAPHITE-REINFORCED T300-W133/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch No.	Ultimate Strength (psi)	Tensile Modulus (10^6psi)	Ultimate Strain (10^{-6}in/in)	Poisson's Ratio
-65°F, Dry	8235 - Avg.	93,190	9.107	10,230	0.060
	Std.Dev.	4,460	0.043	400	---
	8264 - Avg.	89,830	9.229	9,280	0.048
	Std.Dev.	9,700	0.670	530	---
	8284 - Avg.	88,680	9.540	9,210	0.053
	Std.Dev.	2,910	0.281	430	---
72°F, Dry	8235 - Avg.	96,280	8.278	11,160	0.047
	Std.Dev.	3,770	0.258	490	---
	8264 - Avg.	92,240	8.309	10,560	0.054
	Std.Dev.	10,290	0.702	950	---
	8284 - Avg.	89,800	8.987	9,430	0.044
	Std.Dev.	10,870	0.338	1,140	---
200°F, Dry	8235 - Avg.	66,030	8.301	7,827	0.037
	Std.Dev.	5,410	0.387	785	0.020
	8264 - Avg.	78,170	8.596	8,940	0.053
	Std.Dev.	10,230	0.424	992	0.010
	8284 - Avg.	82,250	9.038	8,990	0.082
	Std.Dev.	5,340	0.130	490	---
-65°F, Wet[2]	8235 - Avg.[1]	97,370	9.384	9,760	0.046
	Std.Dev.	4,350	0.369	1,540	---
	8264 - Avg.	94,380	9.510	9,660	0.046
	Std.Dev.	8,530	0.153	1,020	---
	8284 - Avg.	98,220	9.659	10,060	0.068
	Std.Dev.	7,280	0.475	460	---
72°F, Wet	8235 - Avg.	82,990	8.627	9,530	0.037
	Std.Dev.	10,520	0.262	970	---
	8264 - Avg.	89,860	8.794	10,260	0.046
	Std.Dev.	7,080	0.586	870	---
	8284 - Avg.	89,790	8.780	10,290	0.056
	Std.Dev.	5,620	0.197	660	---
200°F, Wet	8235 - Avg.	65,010	7.956	7,060	0.034
	Std.Dev.	5,980	0.703	2,350	0.018(3)
	8264 - Avg.	63,080	8.082	8,160	0.049(4)
	Std.Dev.	2,340	0.701	1,560	0.010
	8284 - Avg.	64,660(5)	7.923(5)	6,979(5)	0.067(3)
	Std.Dev.	4,160	0.583	1,308	0.018

NOTES: (1) All avg. values represent five specimens except Poisson's ratio, which only represents two specimens.

(2) WET = wet aged at 140°F, 100% R.H. until saturated.

(3) Average of three specimens.

(4) Average of four specimens.

(5) Average of six specimens.

TABLE 49

LONGITUDINAL (WARP) COMPRESSIVE PROPERTIES OF GLASS-REINFORCED E-7781/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch Number	Ultimate Strength (psi)	Compressive Modulus (10^6 psi)	Ultimate Strain (10^{-6} in/in)
72°F, Dry	8235 - Avg.[1]	48,800	3.635	15,090
	Std.Dev.	3,750	0.334	2,570
	8264 - Avg.	49,800	3.724	13,360
	Std.Dev.	3,440	0.197	1,200
	8284 - Avg.	50,300	3.690	15,600
	Std.Dev.	1,400	0.120	930
72°F, Wet[2]	8235 - Avg.	23,770	3.070	8,520
	Std.Dev.	3,970	0.298	1,300
	8284 - Avg.	25,450	3.419	7,740
	Std.Dev.	1,880	0.350	1,860
200°F,Wet[2,3]	8235 - Avg.	13,860	3.049	5,230
	Std.Dev.	1,660	0.202	900

NOTES: (1) All avg. values represent five specimens.

(2) WET = wet aged at 140°F/100% R.H. until saturated.

(3) Tested at incorrect temperature. Should have been tested at 72°F.

TABLE 50

TRANSVERSE (FILL) COMPRESSIVE PROPERTIES OF
GLASS-REINFORCED E-7781/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch No.	Ultimate Strength (psi)	Compressive Modulus (10^6 psi)	Ultimate Strain (10^{-6} in/in)
-65°F, Dry	8235 - Avg.	64,140	4.013	17,510
	Std.Dev.	4,650	0.099	1,000
	8264 - Avg.	57,580	4.187	14,620
	Std.Dev.	1,420	0.346	950
	8284 - Avg.	69,750	4.350	15,120
	Std.Dev.	3,800	0.090	1.760
72°F, Dry	8235 - Avg.	41,690	3.788	11,670
	Std.Dev.	3,420	0.138	2,120
	8264 - Avg.	39,080	3.590	11,730
	Std.Dev.	3,020	0.195	2,050
	8284 - Avg.	41,800	3.600	12,250
	Std.Dev.	2,700	0.350	3,320
200°F, Dry	8235 - Avg.	27,950(4)	3.471(4)	9,240(4)
	Std.Dev.	320	0.083	170
	8264 - Avg.	25,040	3.353	7,450
	Std.Dev.	1,610	0.277	1,080
	8284 - Avg.	33,430	3.622	9,630
	Std.Dev.	2,390	0.226	1,880
-65°F, Wet[2]	8235 - Avg.	47,070	4.177	11,900
	Std.Dev.	4,590	0.083	1,790
	8264 - Avg.	43,190	3.881	11,330
	Std.Dev.	3,930	0.342	1,090
	8284 - Avg.	49,990	4.227	13,630
	Std.Dev.	3,600	0.215	1,420
72°F, Wet	8235 - Avg.	21,950	3.329	7,110
	Std.Dev.	2,490	0.045	2,110
	8264 - Avg.	(inadvertently tested @ 200°F, wet)		
	Std.Dev.			
	8284 - Avg.	25,680	3.616	8,210
	Std.Dev.	1,840	0.539	820
200°F, Wet	8235 - Avg.	13,850	3.240	4,490
	Std.Dev.	1,100	0.320	520
	8264 - Avg.	13,260	2.803	4,810
	Std.Dev.	1,700	0.396	1,340
	8284 - Avg.	17,370(3)	3.273(3)	4,730(3)
	Std.Dev.	1,020	0.612	1,460

NOTES: (1) All avg. values represent five specimens unless otherwise noted.

(2) WET = wet aged at 140°F/100% R.H. until saturated.

(3) Average of four specimens.

(4) Average of two specimens.

TABLE 51

LONGITUDINAL (WARP) COMPRESSIVE PROPERTIES OF
GRAPHITE-REINFORCED W-133/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch Number	Ultimate Strength (psi)	Compressive Modulus (10^6 psi)	Ultimate Strain (10^{-6} in/in)
72°F, Dry	8235 - Avg.[1]	66,300[2]	7.67[2]	9,627[2]
	Std.Dev.	9,500	1.16	2,860
	8264 - Avg.	72,100	8.73	8,062
	Std.Dev.	4,800	1.13	1,534
	8284 - Avg.	69,900[3]	8.29[3]	9,532[3]
	Std.Dev.	3,100	1.71	3,334
72°F, Wet	8235 - Avg.	57,510	7.50	8,313
	Std.Dev.	8,910	0.59	2,664
	8264 - Avg.	53,650	7.96	7,397
	Std.Dev.	3,010	1.20	1,794
	8284 - Avg.	50,450	7.69	7,730
	Std.Dev.	2,480	1.57	2,350

NOTES: (1) All avg. values represent five specimens unless otherwise specified.

(2) Three specimens.

(3) Four specimens.

TABLE 52

TRANSVERSE (FILL) COMPRESSIVE PROPERTIES OF
GRAPHITE-REINFORCED W-133/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch Number	Ultimate Strength (psi)	Compressive Modulus (10^6 psi)	Ultimate Strain (10^{-6} in/in)
-65°F, Dry	8235 - Avg.[1]	85,900	9.50	9,993
	Std.Dev.	4,700	1.13	1,680
	8264 - Avg.	77,000	8.14	11,615
	Std.Dev.	6,000	0.47	2,264
	8284 - Avg.	86,500	7.89	13,047
	Std.Dev.	6,300	0.43	888
72°F, Dry	8235 - Avg.	60,080	7.47	7,919
	Std.Dev.	4,680	0.99	872
	8264 - Avg.	59,500[3]	8.32[3]	8,148[3]
	Std.Dev.	5,400	0.74	1,714
	8284 - Avg.	63,030	7.88	8,698
	Std.Dev.	3,010	0.69	3,423
200°F, Dry	8235 - Avg.	43,500	8.11	5,342
	Std.Dev.	1,500	0.76	1,055
	8264 - Avg.	37,300	8.16	4,511
	Std.Dev.	2,400	1.33	980
	8284 - Avg.	40,500	7.57	6,217
	Std.Dev.	1,600	0.27	853
-65°F, Wet[2]	8235 - Avg.	77,220	9.18	9,734
	Std.Dev.	5,740	0.67	2,557
	8264 - Avg.	74,560	8.60	10,476
	Std.Dev.	4,570	0.21	1,383
	8284 - Avg.	77,500	8.60	9,337
	Std.Dev.	7,470	0.46	1,954
72°F, Wet	8235 - Avg.	53,060	8.92	5,893
	Std.Dev.	4,680	0.28	848
	8264 - Avg.	49,070	7.75	7,366
	Std.Dev.	1,760	1.12	3,156
	8284 - Avg.	49,980	8.05	6,754
	Std.Dev.	4,690	0.51	1,256
200°F, Wet	8235 - Avg.	24,370[3]	9.70[3]	2,485[3]
	Std.Dev.	3,170	0.81	421
	8264 - Avg.	27,610	8.15	3,783
	Std.Dev.	4,910	0.69	1,064
	8284 - Avg.	32,240[3]	8.50[3]	3,950[3]
	Std.Dev.	3,400	0.91	704

NOTES: (1) All avg. values represent five specimens unless otherwise noted.

(2) WET = wet aged at 140°F/100% R.H. until saturated.

(3) Four specimens.

TABLE 53

IN-PLANE SHEAR PROPERTIES[1] OF GLASS-REINFORCED E-7781/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch Number	In-Plane Shear Strength (psi)	In-Plane Shear Modulus (10^6 psi)	±45° Tensile Modulus (10^6 psi)	±45° Poisson's Ratio
-65°F, Dry	8235 - Avg.	16,670[2]	0.811[2]	2.678[2]	0.646[2]
	Std.Dev.	2,820	0.082	0.252	0.050
	8264 - Avg.	17,160[3]	0.914[3]	---	---
	Std.Dev.	2,300	0.070	---	---
	8284 - Avg.	16,780	0.808	2.645	0.638
	Std.Dev.	2,320	0.052	0.159	0.044
72°F, Dry	8235 - Avg.	11,340[2]	0.508[2]	1.886[2]	0.850[2]
	Std.Dev.	1,430	0.034	0.103	0.070
	8264 - Avg.	11,710[5]	0.683[5]	---	---
	Std.Dev.	810	0.108	---	---
	8284 - Avg.	11,150	0.463	1.642	0.775
	Std.Dev.	1,050	0.031	0.094	0.066
200°F, Dry	8235 - Avg.	7,060[2]	0.318[2]	1.187[2]	0.871[2]
	Std.Dev.	1,330	0.037	0.135	0.071
	8264 - Avg.	6,940[2]	0.408[2]	---	---
	Std.Dev.	790	0.080	---	---
	8284 - Avg.	7,420	0.308	1.173	0.905
	Std.Dev.	1,380	0.058	0.219	0.038
-65°F, Wet	8235 - Avg.	8,740[4]	0.662[4]	2.295[4]	0.734[4]
	Std.Dev.	1,380	0.037	0.112	0.049
	8264 - Avg.	---	---	---	---
	Std.Dev.	---	---	---	---
	8284 - Avg.	8,310	0.664	2.284	0.721
	Std.Dev.	870	0.096	0.319	0.018
72°F, Wet	8235 - Avg.	5,370[4]	0.339[4]	1.241[4]	0.825[4]
	Std.Dev.	740	0.033	0.177	0.099
	8264 - Avg.	---	---	---	---
	Std.Dev.	---	---	---	---
	8284 - Avg.	5,670	0.381	1.378	0.809
	Std.Dev.	790	0.078	0.294	0.095
200°F, Wet	8235 - Avg.	2,710[4]	0.160[4]	0.597[4]	0.900[4]
	Std.Dev.	480	0.047	0.117	0.180
	8264 - Avg.	---	---	---	---
	Std.Dev.	---	---	---	---
	8284 - Avg.	2,700	0.301	1.091	0.835
	Std.Dev.	370	0.104	0.323	0.120

NOTES: (1) All values represent average of five specimens unless otherwise noted.
(2) Average of seven specimens.
(3) Average of six specimens.
(4) Average of eight specimens.
(5) Average of eleven specimens.

TABLE 54

IN-PLANE SHEAR PROPERTIES OF GRAPHITE-
REINFORCED W133/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch Number	In-Plane Shear Strength (psi)	In-Plane Shear Modulus (10^6 psi)	±45° Tensile Modulus (10^6 psi)	±45° Poisson's Ratio
-65°F, Dry	8235 - Avg.[1]	18,140	0.71	2.54	0.782
	Std.Dev.	2,440	0.08	0.27	0.046
	8264 - Avg.	17,480	0.79	2.76	0.740
	Std.Dev.	1,270	0.09	0.35	0.068
	8284 - Avg.	19,550	0.79	2.79	0.768
	Std.Dev.	620	0.04	0.19	0.053
72°F, Dry	8235 - Avg.	12,950	0.50	1.88	0.893
	Std.Dev.	1,420	0.05	0.21	0.044
	8264 - Avg.	11,900	0.54	1.93	0.795
	Std.Dev.	500	0.07	0.17	0.086
	8284 - Avg.	13,600	0.51	1.93	0.895
	Std.Dev.	1,200	0.05	0.17	0.038
200°F, Dry	8235 - Avg.	7,760	0.32	1.24	0.964
	Std.Dev.	1,080	0.03	0.15	0.052
	8264 - Avg.	7,630	0.37	1.40	0.894
	Std.Dev.	540	0.07	0.23	0.095
	8284 - Avg.	8,060	0.32	1.21	0.911
	Std.Dev.	600	0.04	0.15	0.063
-65°F, Wet[2]	8235 - Avg.	16,300	0.87	2.90	0.673
	Std.Dev.	2,520	0.03	0.23	0.093
	8264 - Avg.	16,830	0.92	3.08	0.669
	Std.Dev.	1,540	0.12	0.41	0.052
	8284 - Avg.	17,280	0.71	2.54	0.791
	Std.Dev.	2,140	0.06	0.26	0.070
72°F, Wet	8235 - Avg.	9,970	0.49	1.70	0.729
	Std.Dev.	1,350	0.05	0.16	0.083
	8264 - Avg.	10,230	0.60	2.13	0.778
	Std.Dev.	1,370	0.12	0.39	0.127
	8284 - Avg.	11,180	0.52	1.95	0.869
	Std.Dev.	970	0.06	0.19	0.108
200°F, Wet	8235 - Avg.	4,530	0.25	0.87	0.755
	Std.Dev.	630	0.06	0.21	0.124
	8264 - Avg.	4,470	0.27	0.99	0.873
	Std.Dev.	500	0.12	0.41	0.186
	8284 - Avg.	4,480	0.23	0.96	1.074
	Std.Dev.	480	0.04	0.12	0.142

NOTES: (1) All values represent average of five specimens unless otherwise noted.
(2) WET = wet aged at 140°F/100% R.H. until saturated.

TABLE 55

INTERLAMINAR SHEAR PROPERTIES OF
EA9396 COMPOSITE MATERIAL

Reinforcement Type	Resin Batch	Test Condition	Shear Str.(1,2) (psi) Mean	Std.Dev.	Wt. Gain During Aging(%)
"E"-7781-Glass	8235	72°F, Dry	5800	240	N.A.
	8264	72°F, Dry	5550	180	N.A.
	8284	72°F, Dry	5510	240	N.A.
	8235	72°F, Wet(3)	2710	50	2.32
	8264	72°F, Wet(3)	3010	120	2.10
	8284	72°F, Wet(3)	3220	50	2.09
	8235	200°F, Wet(3)	1340	70	2.24
	8264	200°F, Wet(3)	1920	80	2.09
	8284	200°F, Wet(3)	1880	80	2.11
T300-W133-Graphite	8235	72°F, Dry	5660	400	N.A.
	8264	72°F, Dry	5780	660	N.A.
	8284	72°F, Dry	5650	340	N.A.
	8235	72°F, Wet(3)	4750	410	2.19
	8264	72°F, Wet(3)	4730	180	2.32
	8284	72°F, Wet(3)	4610	110	2.24
	8235	200°F, Wet(3)	2060	170	2.22
	8264	200°F, Wet(3)	2250	70	2.32
	8284	200°F, Wet(3)	2720	140	2.24

NOTES: (1) Four-point shear. Graphite tests at 16:1 span-thickness ratio, Glass tests at 8:1. All values represent average of five specimens.
(2) Warp direction of fabric is running in length direction of specimens.
(3) Wet = Aging at 140°F, 95-100% R.H. until saturated.

TABLE 56

EFFECT OF 350°F CURE CYCLES ON INTERLAMINAR SHEAR STRENGTH OF EA9396 COMPOSITE LAMINATES

Reinforcement Type	Exposure Condition	Test Condition	Shear Strength (psi)(1,2,3) Mean	Std.Dev.	Wt. Gain During Aging (%)
"E"-7781-Glass	2 hrs @ 350°F	72°F, Dry	5650	330	N.A.
	2 hrs @ 350°F	200°F,Wet(4)	1930	120	2.04
	16 hrs @ 350°F (3 segments of 6 + 6 + 4 hrs)	72°F, Dry	6010	180	N.A.
		200°F,Wet(4)	1930	150	2.25
T300-W133-Graphite	2 hrs @ 350°F	72°F, Dry	5030	210	N.A.
	2 hrs @ 350°F	200°F,Wet(4)	1990	120	2.36
	16 hrs @ 350°F (3 segments of 6 + 6 + 4 hrs)	72°F, Dry	5260	220	N.A.
		200°F,Wet(4)	1920	120	2.35

NOTES:
(1) Four-point shear (Graphite tests at 16:1 span-thickness ratio, Glass tests at 8:1).
(2) Warp direction of fabric is running in length direction of specimens.
(3) The glass reinforced specimens turned very noticeably darker after this exposure. This could not be noticed on the black graphite reinforced specimens.
(4) Wet = aging at 140°F, 95-100% R.H. until saturated.

TABLE 57

ROOM TEMPERATURE BEARING STRENGTH OF EA9396 COMPOSITE MATERIAL

Reinforcement	Aging Condition	Bearing Strength @ 4% Elong. (ksi) Avg.	S.D.	Max. Bearing Strength (ksi) Avg.	S.D.
E/7781 Glass	None (dry)	42.4	1.1	54.9	2.7
	Wet[1]				
T300/W133 Graphite	None (dry)	58.3	12.4	71.3	6.8
	Wet[1]				

NOTE: [1]Wet aged at 140°F, 95-100% R.H. until saturated.

TABLE 58

GLASS TRANSITION TEMPERATURE OF EA9396
NEAT RESIN CASTINGS

Resin Batch Number	Test Condition	Tg (°C)	Tg (°F)	Weight Gain During Aging (%)
8235 - Avg.	Dry	175	347	N.A.
Std.Dev.		1	2	
8264 - Avg.	Dry	176	349	N.A.
Std.Dev.		1	2	
8284 - Avg.	Dry	177	350	N.A.
Std.Dev.		0.3	0.5	
8235 - Avg.	Wet (1)	106	223	9.1
Std.Dev.		0.6	1	
8264 - Avg.	Wet (1)	108	226	8.7
Std.Dev.		2.5	4.5	
8284 - Avg.	Wet (1)	107	225	8.8
Std.Dev.		2	3.6	

NOTE: (1) Wet = Aging at 140°F, 95-100% R.H. until saturated.

TABLE 59

EFFECT OF RESIN/FIBER CONTENT ON INTERLAMINAR SHEAR STRENGTH
OF EA9396 COMPOSITE LAMINATES

Reinforcement Type	Fiber Content (Vol. %) Target	Actual	Resin Content (Wt. %) Target	Actual	Void Content (Vol. %)	72°F, Dry Shear Strength (psi) (1) Mean	Std. Dev.	No. Tests	72°F, Wet Shear Strength (psi) (1) Mean	Std. Dev.	No. Tests
"E"-7781-Glass	65	55.4	18	26.2	5.0	5290	40	3	3110	200	2
	55	54.3	27	26.7	6.1	5540	80	3	3180	20	2
	55	54.0	27	26.5	6.9	5930	400	5	2710	50	5
	45	45.6	35	35.2	4.7	5500	120	4	---	---	---
	45	52.3	35	28.4	6.4	5490	620	3	2970	740	2
	35	48.1	45	31.9	6.7	4670	50	3	2450	110	2
T300-W133-Graphite	65	55.8	24	32.5	6.5	5870	140	3	4290	30	2
	55	54.6	34.7	35.6	4.0	5950	390	5	4750	410	5
	55	54.7	34.7	33.6	6.4	5860	440	3	4740	160	2
	45	50.1	43.7	39.0	4.9	6320	230	3	4960	275	2
	35	49.8	48	39.0	4.7	6350	190	3	5120	5	2

NOTE: (1) Four-point shear. Graphite tests at 16:1 span-thickness ratio, Glass tests at 8:1.

TABLE 60

VISCOSITY BEHAVIOR OF EA9396 PARTS A AND B
AFTER EXTENDED STORAGE PERIODS

Storage Conditions	Viscosity (poise) (1)	
	Part A(2)	Part B(3)
Initial	840	1
1 month at 100°F	840	0.75
1 month at 120°F	880	0.75
3 months at 72°F	920	1
6 months at 72°F	720	1
8 months at 100°F	840	1
8 months at 120°F	880	1
12 months at 72°F	720	1
12 months at 100°F	960	1
12 months at 120°F	1000	1

NOTES: (1) Viscosity measured at 72°F.
(2) Brookfield, spindle #7.
(3) Brookfield, spindle #3.

TABLE 61

CALORIMETRIC CURE CHARACTERISTICS OF EA9396 RESIN
AFTER EXTENDED STORAGE PERIODS

Storage Conditions	Temperature at Reaction Peak (°C)	Total Heat of Cure (J/gm)
Initial	112.5	576.2
1 month at 100°F	115.2	588.8
1 month at 120°F	114.6	576.4
3 months at 72°F	115.0	572.9
6 months at 72°F	108.9	633.8
6 months at 100°F	110.9	593.5
6 months at 120°F	111.8	624.6
12 months at 72°F	112.8	613.4
12 months at 100°F	114.3	589.5
12 months at 120°F	112.6	601.6

NOTE: Dynamic DSC test at heating rate of 10°C/minute under nitrogen.

TABLE 62

VISCOSITY PROFILE CHARACTERISTICS OF EA9396
RESIN AFTER EXTENDED STORAGE PERIODS

Storage Conditions	Temperature at Minimum Viscosity (°C)	Minimum Viscosity (poise)	Gel Temperature (°C)
Initial	49	~1000	70.5
1 month at 100°F	51	~150	71.6
1 month at 120°F	47	~150	71.9
3 months at 72°F	50	~40	73.3
6 months at 72°F	48	~110	66.8
6 months at 100°F	45	~200	65.9
6 months at 120°F	44	~250	67.8
12 months at 72°F	67	~149	71.9
12 months at 100°F	66	~46	72.6
12 months at 120°F	64	~64	71.4

NOTE: Test conducted on Rheometrics Solids Analyzer using a heating rate of 1°C/minute.

TABLE 63

EFFECT OF EXTENDED STORAGE ON INTERLAMINAR SHEAR
STRENGTH OF EA9396/GRAPHITE (T300-W133) COMPOSITES

Storage Conditions	Interlaminar Shear Strength (psi)	
	72°F, Dry	72°F, Wet (1)
Initial	5450	3760
1 month at 100°F	6170	4760
1 month at 120°F	5700	4330
3 months at 72°F	5520	4560
6 months at 72°F	6180	4410
6 months at 100°F	6200	4690
6 months at 120°F	6220	4250
12 months at 72°F	5360	4340
12 months at 100°F	6340	4750
12 months at 120°F	6170	3780

(1) Wet = wet aged at 140°F/100% R.H. until saturated.

TABLE 64

POT LIFE OBSERVATIONS FOR 200-GRAM BATCH OF EA9396

Test Temperature (°F)	Time to First Notice of Exotherm (min. after mixing)	Time to Boil/Gel[1] (min. after mixing)
72	105	112/113
100	40	60/61

[1]At 112 and 60 minutes, respectively, the two samples were so hot that they were bubbling and smoking. Gel followed within a minute of this.

TABLE 65

POT LIFE OBSERVATIONS OF 12-PLY GRAPHITE LAMINATE IMPREGNATED WITH EA9396 A/B RESIN

Temperature Environment (°F)	Maximum Midplane Temperature[1] (°F)	Time to Become Tacky[2] (min)	Time to Lose Tack[3] (min)	Time at Which Surface Is no Longer Indentable[4] (min)
72	84	~60	210	240
100	116	~50	100	100

[1]Midplane temperature recorded with imbedded thermocouple.

[2]Subjective judgement of when resin changes from thick syrupy state to a sticky, non-liquid state.

[3]Subjective judgement.

[4]Unable to make an impression on surface of laminate with a wooden tongue depressor and hand pressure.

TABLE 66

EFFECT OF CURE PRESSURE ON INTERLAMINAR SHEAR AND PHYSICAL PROPERTIES OF GLASS-REINFORCED E-7781/EA9396 COMPOSITE LAMINATES

Physical Properties					
Vacuum Level During Cure(1)	Cured Composite Specific Gravity	Resin Content (o/w)	Fiber Content (o/v)	Void Content (o/v)	Ply Thickness (mils)
10" Hg	1.87	28.6	52.6	5.4	8.5
16" Hg	1.88	25.6	55.0	7.1	8.3
23" Hg	1.91	24.4	56.6	6.7	8.0
27" Hg	1.94	26.2	56.1	3.9	7.8

Mechanical Properties					
Vacuum Level During Cure(1)	Test Condition	Span:Thickness Ratio	Interlaminar Shear Strength (psi)	No. of Specimens	Failure Mode
10" Hg	72°F	16	4510	2	Compr.
		8	4640	2	
	200°F	16	2790	3	Compr.
		8	3650	1	
16" Hg	72°F	16	3710	2	Shear
		8	5170	2	Shear
	200°F	16	2340	2	Shear&
			5550	3	Compr.
		8			Shear
23" Hg	72°F	16	3950	3	Shear & Compr.
		8	4630	3	Shear
	200°F	16	2450	2	Shear & Compr.
		8	4740	2	Shear
27" Hg	72°F	16	N.A.	---	---
		8	5940	3	Shear
	200°F	16	N.A.	---	---
		8	3940	2	Shear

NOTE: (1) Cured at 200°F for 45 minutes under vacuum pressure.

TABLE 67

LONGITUDINAL (WARP) TENSILE PROPERTIES OF A1100-SIZED GLASS REINFORCED E-7781/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch Number	Ultimate Strength (psi)	Tensile Modulus (10^6psi)	Ultimate Strain (10^{-6}in/in)	Poisson's Ratio
72°F, Dry	8284 - Avg.[1]	40,760	3.55	11,880	0.104
	- Std.Dev.	3,670	0.17	1,350	0.002
72°F, Wet[2]	8284 - Avg.[1]				
	- Std.Dev.				
200°F, Wet[2]	8284 - Avg.[1]				
	- Std.Dev.				

NOTES: (1) All average values represent five specimens.

(2) WET = wet aged at 140°F/100% R.H. until saturated.

TABLE 68

IN-PLANE SHEAR PROPERTIES OF A1100-SIZED GLASS REINFORCED E-7781/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch Number	In-Plane Shear Strength (psi)	In-Plane Shear Modulus (10^6psi)	±45° Tensile Modulus (10^6 psi)	Poisson's Ratio
72°F, Dry	8284 - Avg.[1]	9,520	0.635	1.93	0.522
	- Std.Dev.	830	0.023	0.14	0.084
72°F, Wet[2]	8284 - Avg.[1]				
	- Std.Dev.				
200°F, Wet[2]	8284 - Avg.[1]				
	- Std.Dev.				

NOTES: (1) All values represent average of five specimens.

(2) WET = wet aged at 140°F/100% R.H. until saturated.

TABLE 69

INTERLAMINAR SHEAR PROPERTIES OF A1100-SIZED GLASS REINFORCED E-7781/EA9396 COMPOSITE MATERIAL

Test Condition	Resin Batch Number	Interlaminar Shear Strength (psi)	Weight Gain During Aging (%)
72°F, Dry	8284 - Avg.[1]	6,150	N.A.
	- Std.Dev.	190	---
72°F, Wet[2]	8284 - Avg.[1]		
	Std.Dev.		
200°F, Wet[2]	8284 - Avg.[1]		
	- Std.Dev.		

NOTES: (1) All values represent average of five specimens.

(2) WET = wet aged at 140°F/100% R.H. until saturation.

TABLE 70

PHYSICAL AND MECHANICAL PROPERTIES OF RTM AND PRESS CURED FIBERGLASS/EPOXY COMPOSITES

Processing Procedure	No. Reinforcing Plies		
	4 ply	5 ply	6 ply
Resin Transfer Molding[3]			
Short Beam Shear Str.(psi)[1]	5613	6358	6321
Flexure Strength[1] (psi)	72,300	74,500	75,800
Flexure Modulus(10^6 psi)[1]	3.034	3.726	4.072
Resin Content (wt. %)[2]	38.8	32.7	27.9
Fiber Content (vol. %)[2]	40.7	46.8	51.5
Voids (vol. %)[2]	3.7	4.7	5.8
Thickness (inch)[1]	0.126	0.131	0.146
Ply Thickness (inch)[1]	0.0315	0.0262	0.0243
Press Cure[4]			
Short Beam Shear Str.(psi)[1]	5544	6282	5899
Flexure Strength[1] (psi)	73,200	67,400	64,100
Flexure Modulus(10^6 psi)[1]	3.634	4.015	3.555
Resin Content (wt. %)[2]	30.2	26.3	30.7
Fiber Content (vol. %)[2]	50.2	53.2	47.2
Voids (vol. %)[2]	5.3	6.1	8.6
Thickness (inch)[1]	0.101	0.116	0.156
Ply Thickness (inch)[1]	0.0253	0.0232	0.0260

[1]Average of five samples.

[2]Average of nine samples.

[3]Mold preheated to 200°F. Resin injected under pressure of 60 psi. Cured for 30 min. at 200°F.

[4]Wet layup cured in closed mold at 200°F and 15.6 psi pressure for 30 minutes.

TABLE 71

BURST PRESSURE OF WET WOUND AND RESIN TRANSFER MOLDED PRESSURE VESSELS

Bottle No.	Impregnation Technique	Burst Pressure (psi)
1	Wet Wound	2800
2	Wet Wound	2100
3	Wet Wound	Not Recorded
4	Resin Transfer Molded	1000[1]

[1]Maximum pressure obtained.

TABLE 72

PHYSICAL PROPERTIES OF PRESSURE VESSELS

Bottle Number	Section	Density (gms/cc)	Fiber Content (% vol.)	Resin Content (% wt.)	Void Content (% vol.)
1	Hoop	1.47	61.9	27.0	3.1
1	Dome	1.47	58.5	31.1	1.1
2	Hoop	1.50	63.4	27.0	0.7
2	Dome	1.50	61.3	29.2	0.2
3	Hoop	1.45	56.8	32.4	1.4
3	Dome	1.47	58.5	31.2	1.4
4	Hoop	1.57	68.9	23.8	0
4	Dome	1.56	68.3	24.3	0

TABLE 73

FLEXURE STRENGTHS OF
XP2942/310 CARBON COMPOSITES

Test Temperature	Flexure Strength (10^3 psi)			
	Batch No. 1		Batch No. 2	
	PPI	UDRI	PPI	UDRI
R.T.	298.7	193.9	240.0	198.5
450°F	298.7	34.6	---	73.9
600°F	285.3	25.7	180.7	48.6
700°F	---	---	125.5	---

TABLE 74

TEST PROGRAM VARIABLES

Materials

1. Cast and stretched acrylic and polycarbonate.
2. Various constructions (different ply materials).
3. Applicability to various curvatures, transparency thicknesses, materials, construction types, and coatings.

Equipment Parameters

4. Various power settings.
5. Various susceptor types.

Technique Evaluation

6. Evaluate patches for visual appearance and leaks.
7. Demonstrate that each selected repair can withstand 10 pressure cycles.

TABLE 75

TEST PROGRAM VARIABLES

Materials

1. Survey candidate adhesives compatible with various transparent materials, coatings, types, etc.
2. Evaluate promising candidates for repair processability. Important factors are process simplicity, shelf life, work life, minimum time to complete cure, surface preparation requirements, and equipment needed.

Technique Evaluation

3. Evaluate patch for visual appearance and leaks.
4. Demonstrate that each selected repair can withstand 10 pressure cycles.

TABLE 76

POLYCARBONATE CANOPY

		FUSION BONDING		ADHESIVE BONDING	
PATCH	PRE-FORM	LARGE PATCH	SMALL PATCH	LARGE PATCH	SMALL PATCH
CA (3/8")	HEAT				
	VACUUM				
	NONE				
CA (1/8")	HEAT				
	VACUUM				
	NONE				
CAB (1/16")	HEAT				
	VACUUM				
	NONE				

/// = PASSED TEST

= PASSED TEST BUT NOT RECOMMENDED

▒▒▒ = FAILED TEST

TABLE 77

CAST ACRYLIC CANOPY

		FUSION BONDING		ADHESIVE BONDING	
PATCH	PRE-FORM	LARGE PATCH	SMALL PATCH	LARGE PATCH	SMALL PATCH
CA (3/8")	HEAT				
	VACUUM				
	NONE				
CA (1/8")	HEAT				
	VACUUM				
	NONE				
CAB (1/16")	HEAT				
	VACUUM				
	NONE				

\\\ = PASSED TEST

= PASSED TEST BUT NOT RECOMMENDED

::: = FAILED TEST

TABLE 78

STRETCHED ACRYLIC CANOPY

		FUSION BONDING		ADHESIVE BONDING	
PATCH	PRE-FORM	LARGE PATCH	SMALL PATCH	LARGE PATCH	SMALL PATCH
CA (3/8")	HEAT				
	VACUUM				
	NONE				
CA (1/8")	HEAT				
	VACUUM				
	NONE				

//// = PASSED TEST

= PASSED TEST BUT NOT RECOMMENDED

= FAILED TEST

= IMPLIES FAILED LEAK TEST, BUT WITH ADDITIONAL SEALANT APPLIED, THIS TECHNIQUE IS RECOMMENDED

TABLE 79

EFFECT OF QUV AGING ON OPTICAL PROPERTIES OF COATED POLYCARBONATE SAMPLES FROM EPOLIN

Specimen Number	QUV Exposure Level	% Transmittance	% Haze
A	0	85.3	2.51
	1 year	80.1	6.35
	2 year	78.1	9.76
	3 year	77.0	6.50
B	0	82.5	2.34
	1 year	79.7	4.34
	2 year	78.6	8.45
	3 year	80.3	6.88
C	0	78.3	15.6
	1 year	76.5	18.3
	2 year	73.5	24.8
	3 year	75.0	24.2
D	0	82.7	1.44
	1 year	83.0	4.25
	2 year	81.7	7.18
	3 year	81.1	6.49
E	0	77.7	6.96
	1 year	77.0	10.51
	2 year	74.8	12.83
	3 year	74.8	18.0

TABLE 80

EFFECT OF QUV AGING ON TAPE ADHESION PERFORMANCE OF COATED POLYCARBONATE SAMPLES FROM EPOLIN

Specimen Number	QUV Exposure Level	Surface[1]	Tape Adhesion Result
A	1 year	Front	No effect
		Rear	No effect
	2 year	Front	No effect
		Rear	No effect
	3 year	Front	16% removal
		Rear	No effect
B	1 year	Front	No effect
		Rear	No effect
	2 year	Front	No effect
		Rear	No effect
	3 year	Front	No effect
		Rear	No effect
C	1 year	Front	No effect
		Rear	6% removal
	2 year	Front	No effect
		Rear	No effect
	3 year	Front	No effect
		Rear	No effect
D	1 year	Front	No effect
		Rear	No effect
	2 year	Front	No effect
		Rear	No effect
	3 year	Front	No effect
		Rear	No effect
E	1 year	Front	No effect
		Rear	No effect
	2 year	Front	No effect
		Rear	No effect
	3 year	Front	No effect
		Rear	No effect

[1]Front surface faced inward in QUV cabinet. Rear surface faced outward in QUV cabinet.

TABLE 81

ARM-100/ELASTOMER SEAL DATA

Property	Eypel (302°F)**		Fluorosilicone (300°F)		NBR-H/160°F (High AN*)		NBR-L/160°F (Low AN*)		Fluoroelast. (350°F)		Chloroprene (250°F)	
	Orig.	Aged	Orig.	Aged	Orig.	Aged	Orig.	Aged	Orig.	Aged	Orig.	Aged
Tensile Str. (psi,	1630	1195	1160	435	3085	2465	2890	1785	2125	1165	3045	1780
Elongation, %	160	200	205	120	440	290	315	160	220	140	315	165
Hardness	70	57	62	57	69	53	72	49	72	61	70	59
Volume Swell (%)	---	10.2	---	6.4	---	29.5	---	76.5	---	18.7	---	17.5

* AN = Acrylonitrile

** Elastomer/aging temperature (70 hrs.)

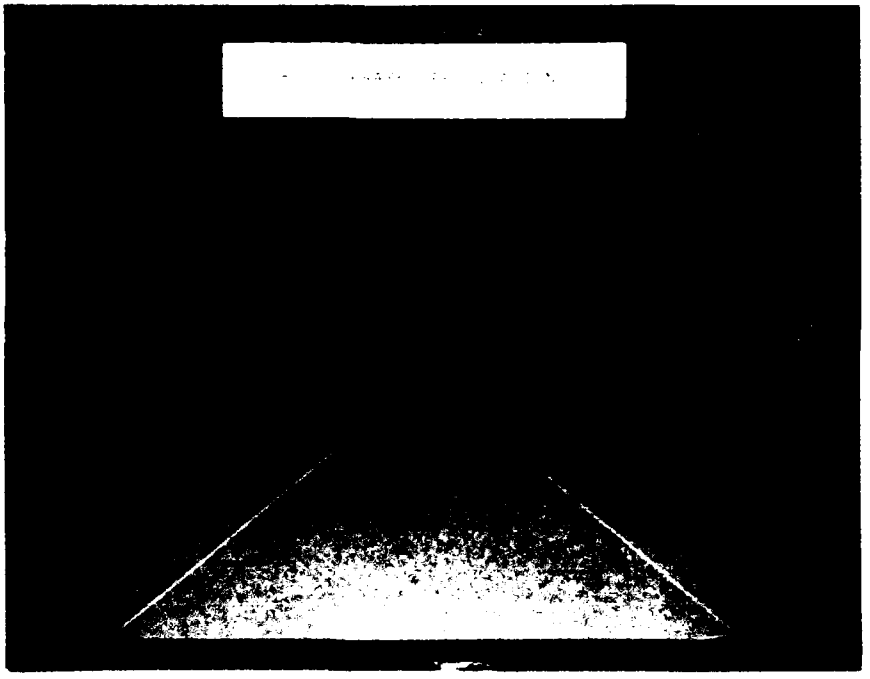
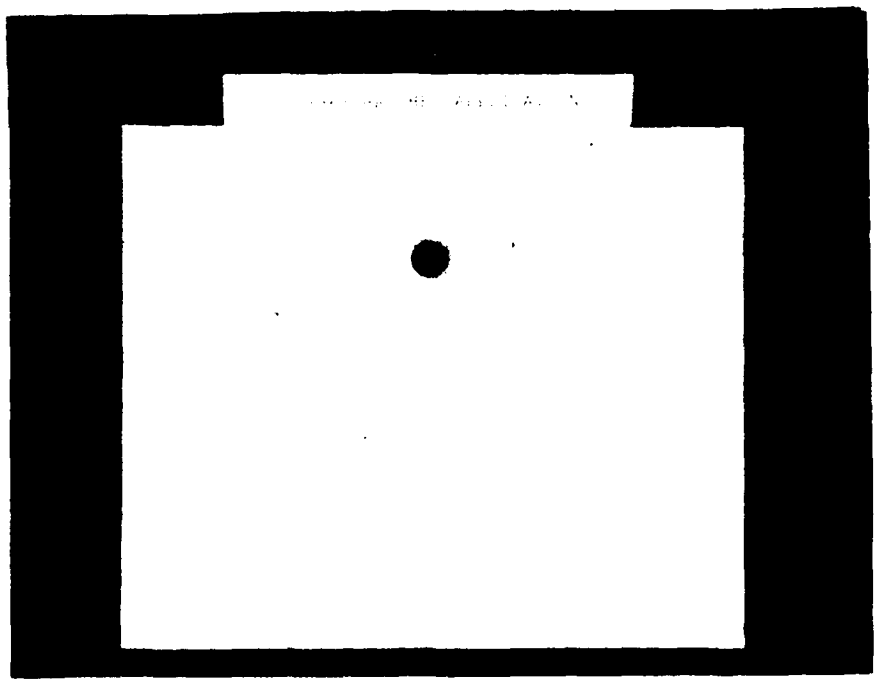
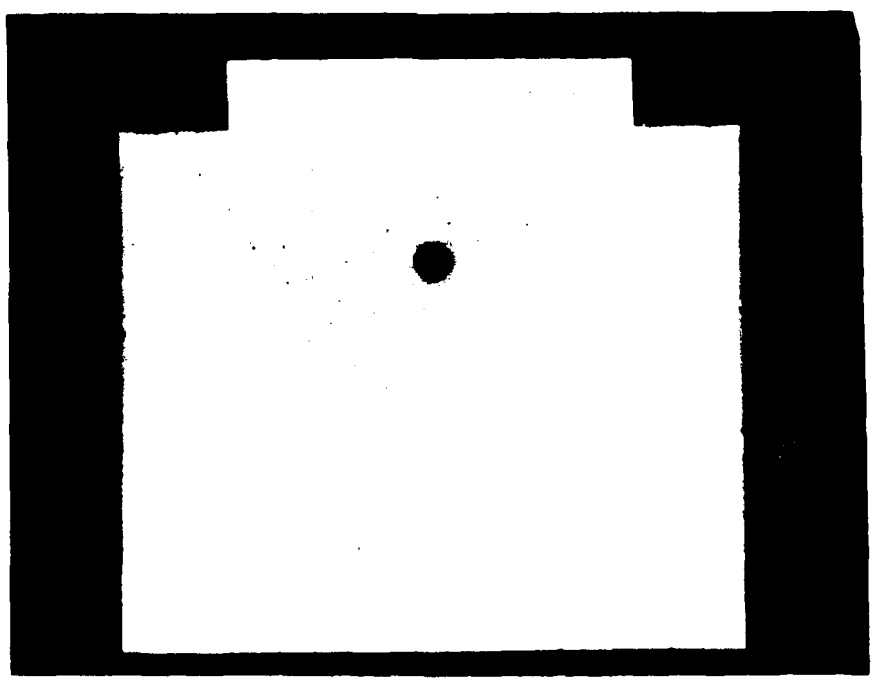


Figure 1. R-500 Primed Aluminum Panel Before Salt Fog Aging.

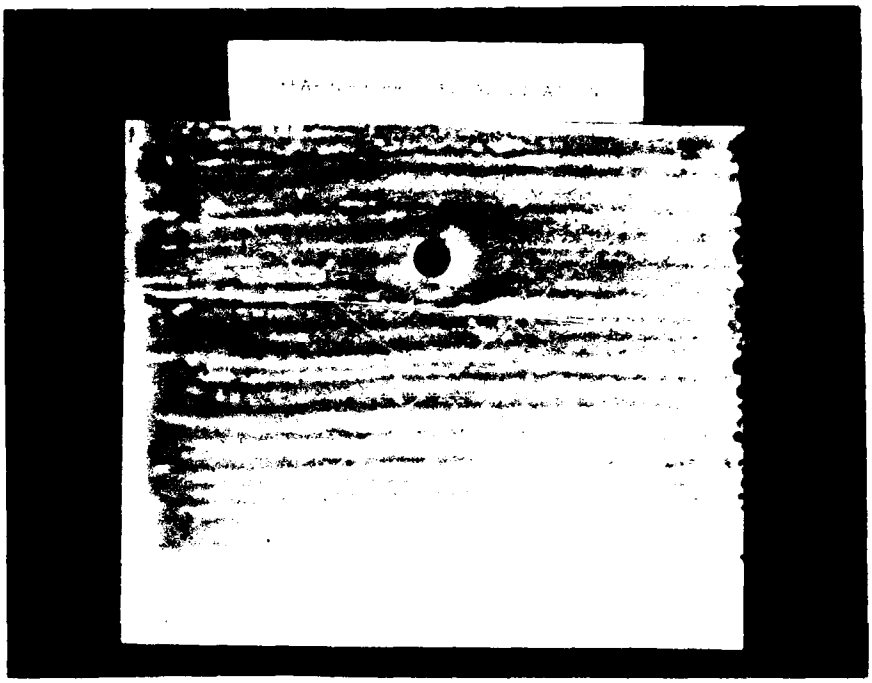


(a) Brushed

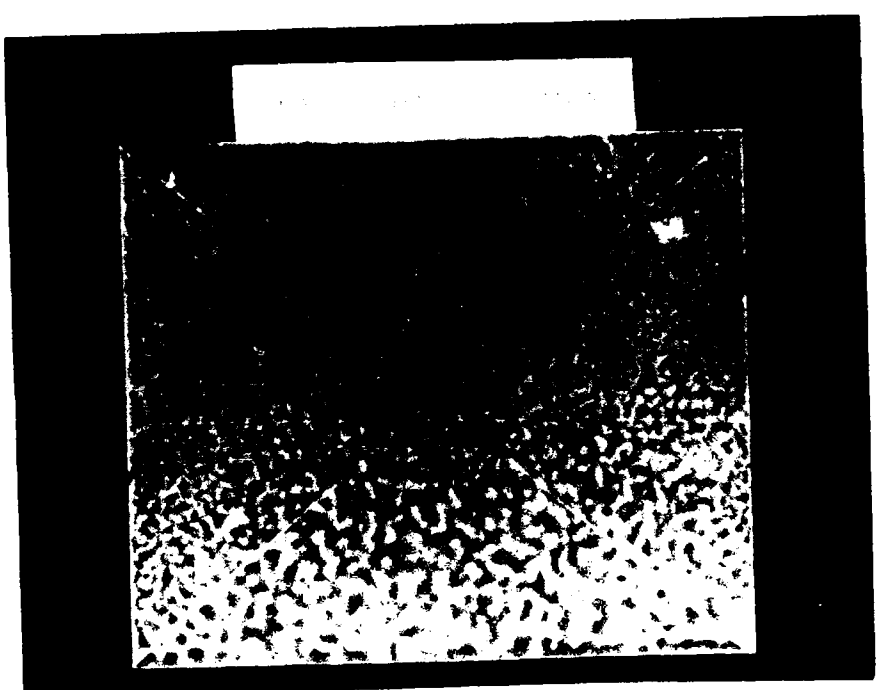


(b) Sprayed

Figure 2. EC-3983 Primed Aluminum Panel Before Salt Fog Aging.



(a) Brushed



(b) Sprayed

Figure 3. XEA-9289 Primed Aluminum Panel Before Salt Fog Aging.

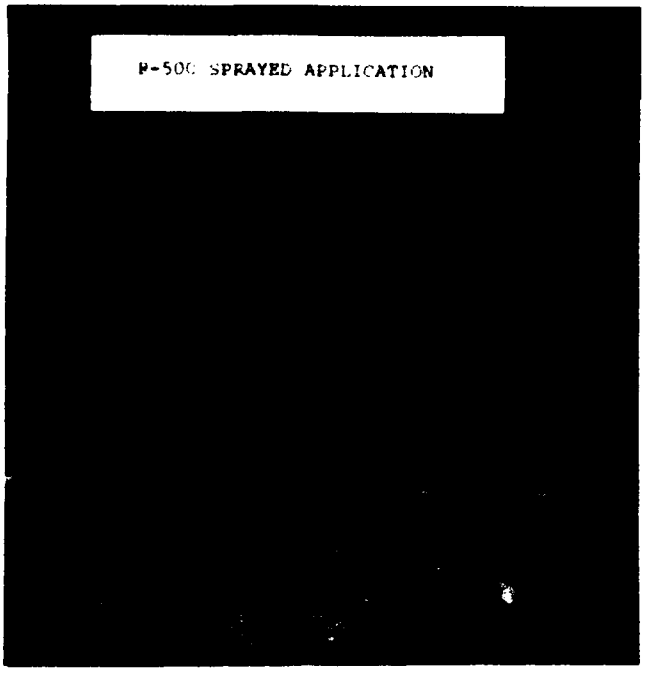
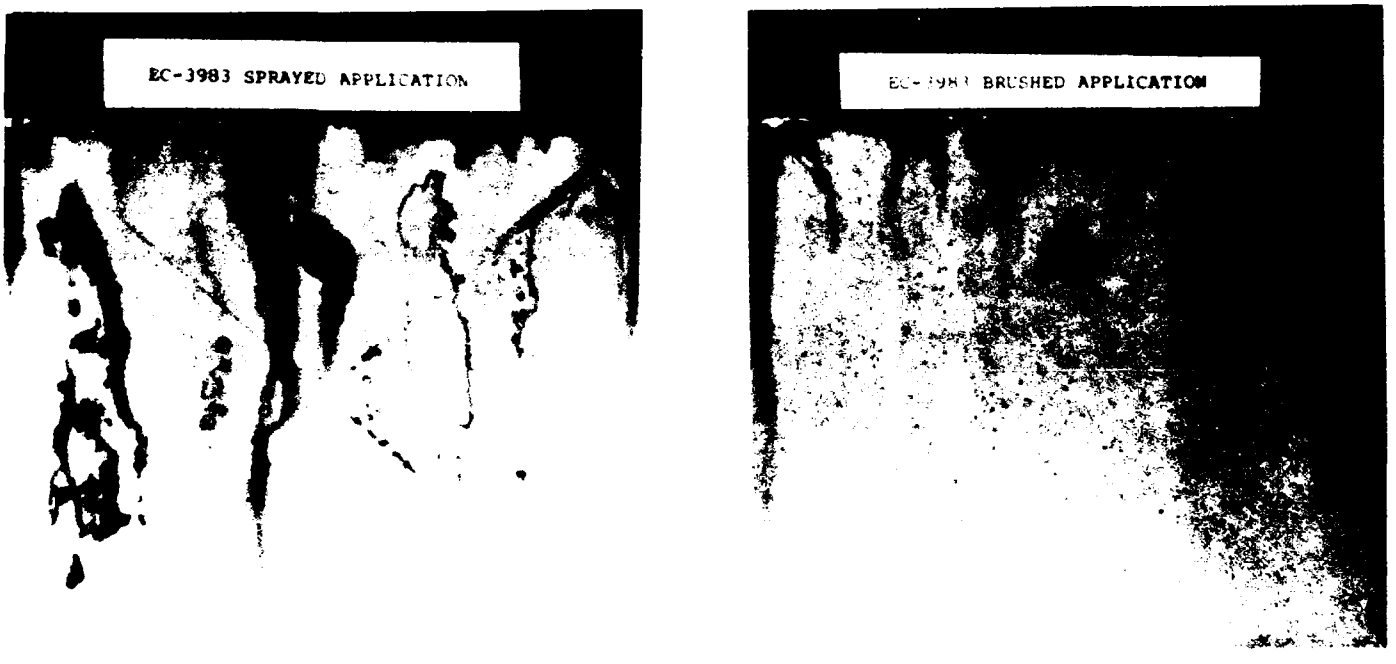
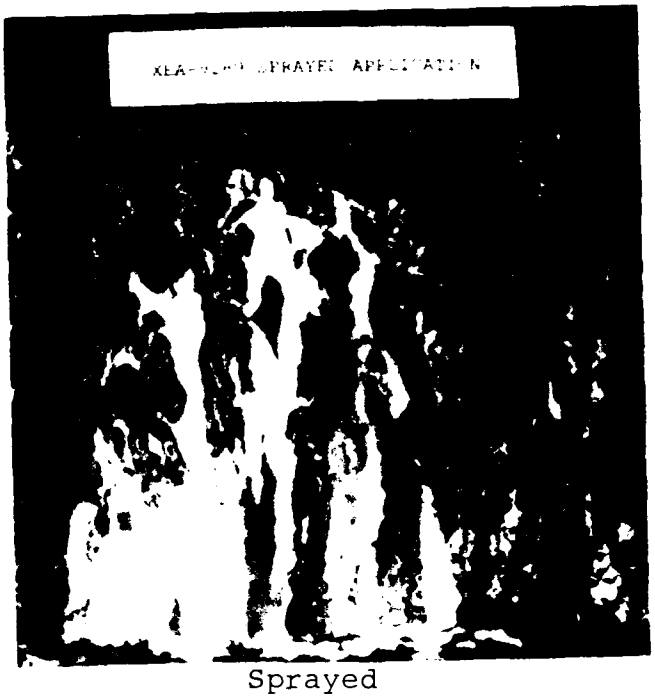


Figure 4. R-500 Primer After 30-Day Exposure to Salt Fog.

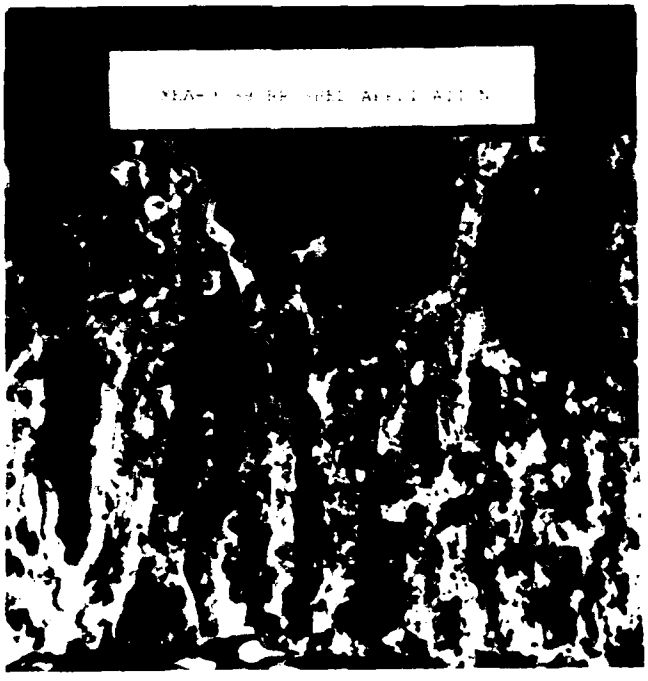


Sprayed Brushed

Figure 5. EC-3983 Primer After 30-Day Exposure to Salt Fog.



Sprayed



Brushed

Figure 6. XEA-9289 Primer After 30-Day Exposure to Salt Fog.

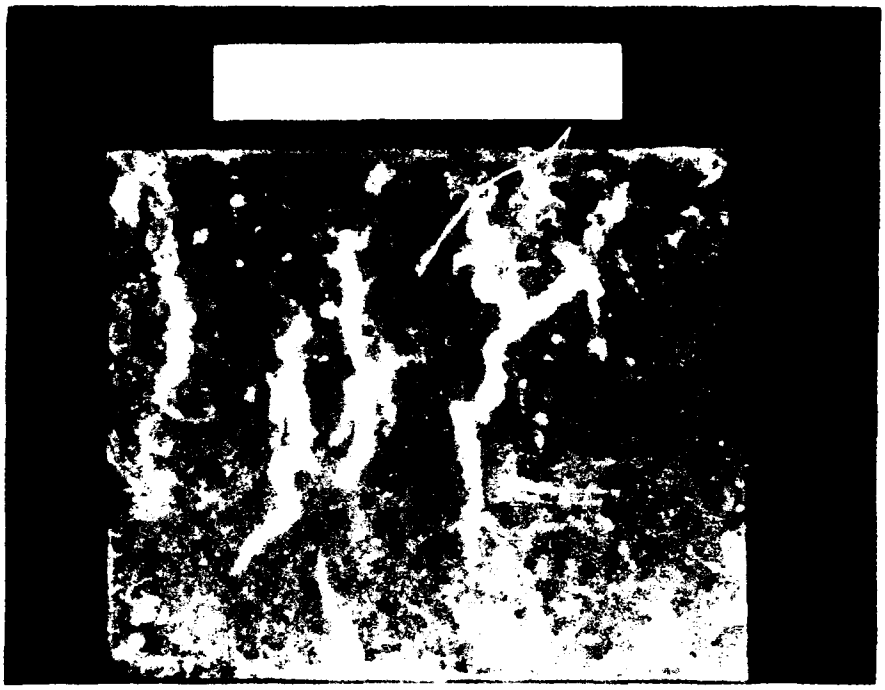


Figure 7. R-500 Primed Panel After 90 Days Exposure to 5% Salt Fog and 95°F.

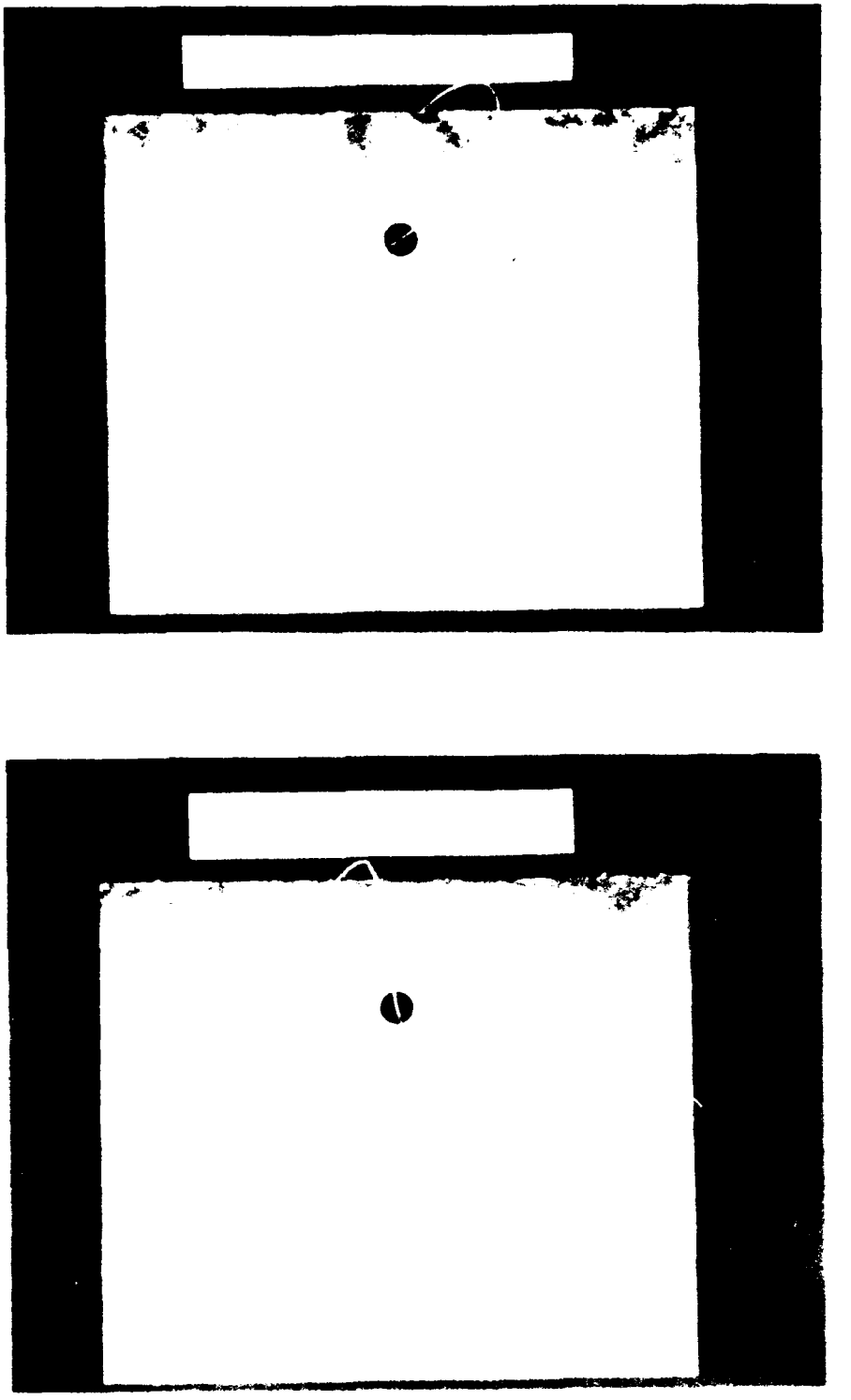


Figure 8. EC-3983 Primed Panel After 90 Days Exposure to 5% Salt Fog and 95°F.

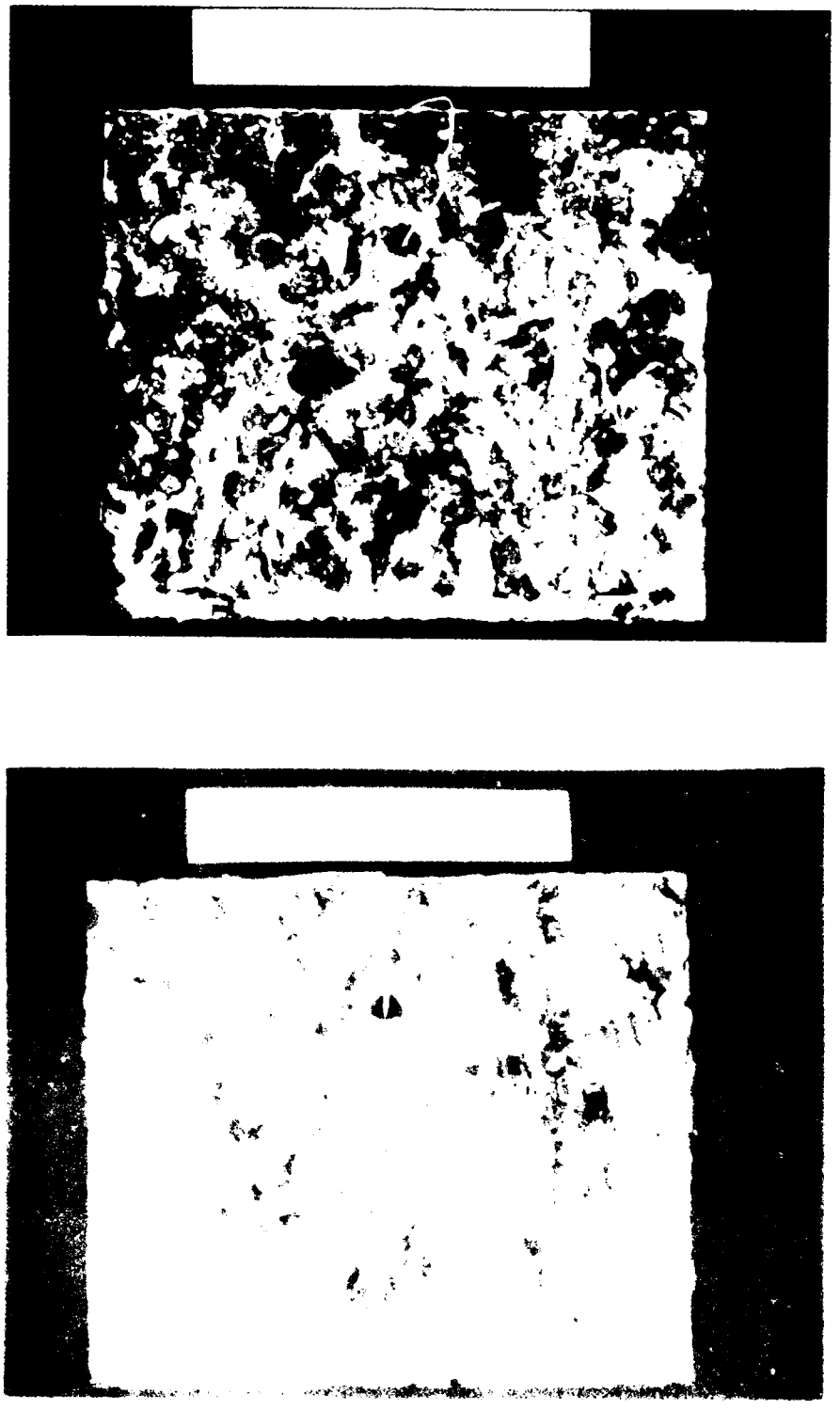


Figure 9. Primed and 250°F Cured XEA-9289 Panel After 90-Day Exposure to 5% Salt Fog and 95°F.

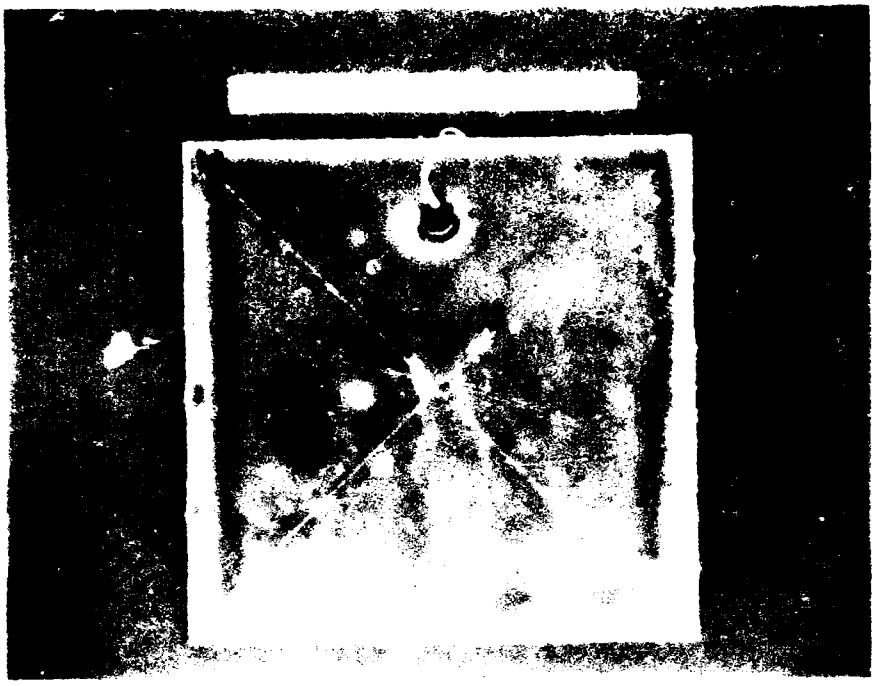


Figure 10. XEA-9289 Primer With a 350°F Cure After 30-Day Exposure to Salt Fog.

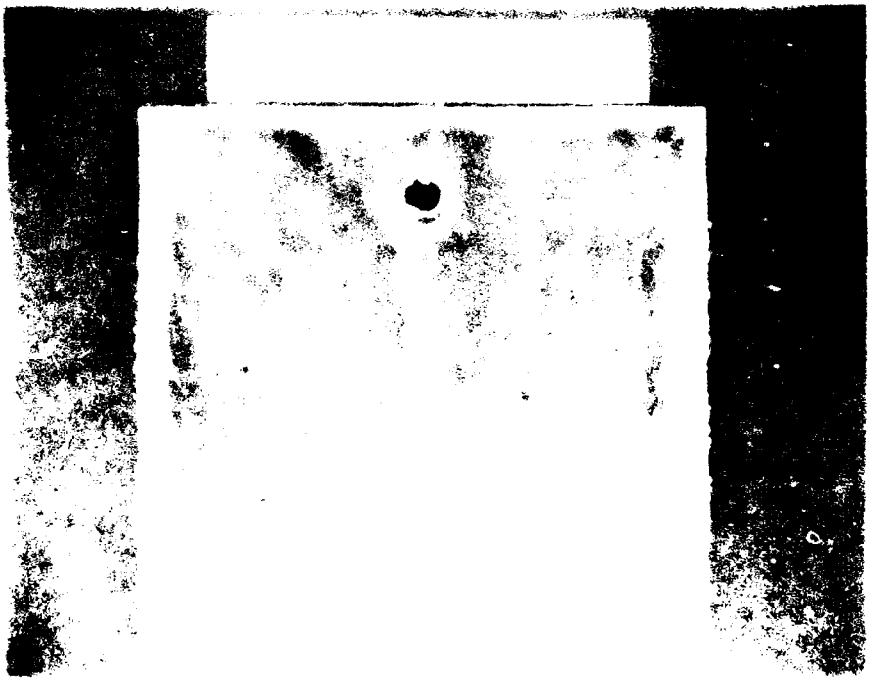
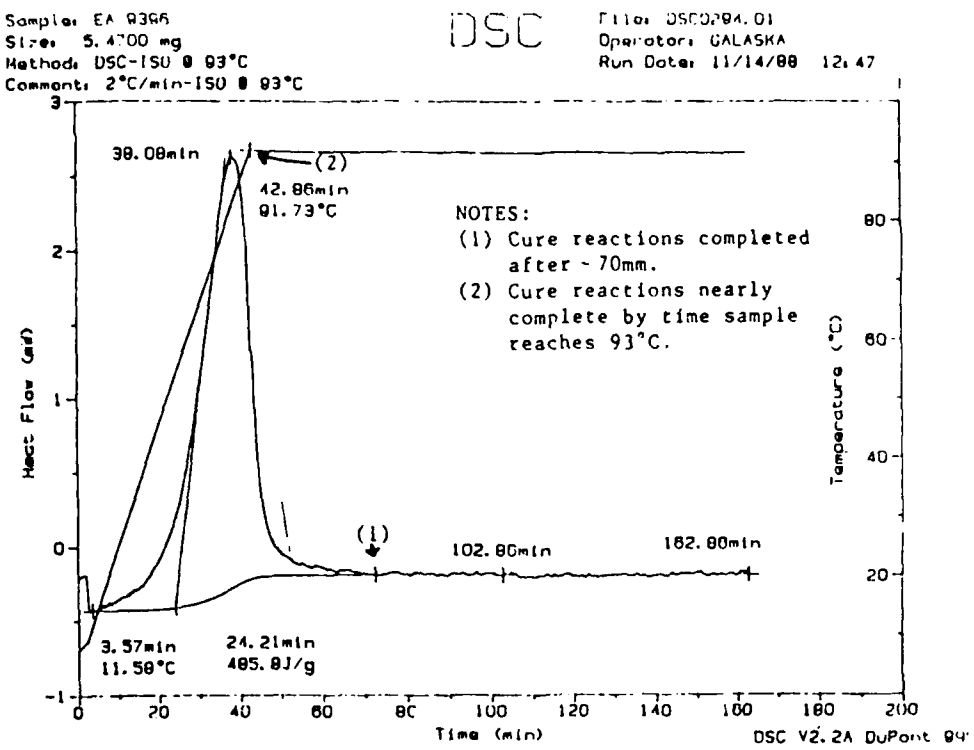
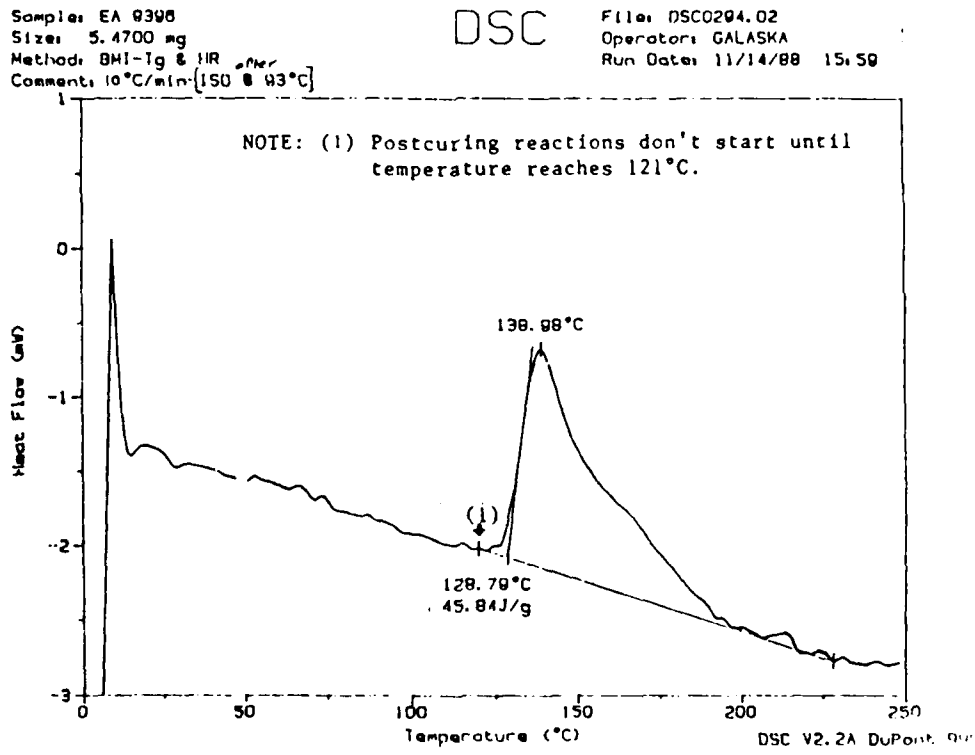


Figure 11. XEA-9289 Primer With a 350°F Cure After 90-Day Exposure to Salt Fog.



(a) Isothermal Cure at 93°C (200°F).



(b) Postcure of Sample Cured in (a).

Figure 12. Cure and Postcure Behavior of EA9396 Epoxy Resin.

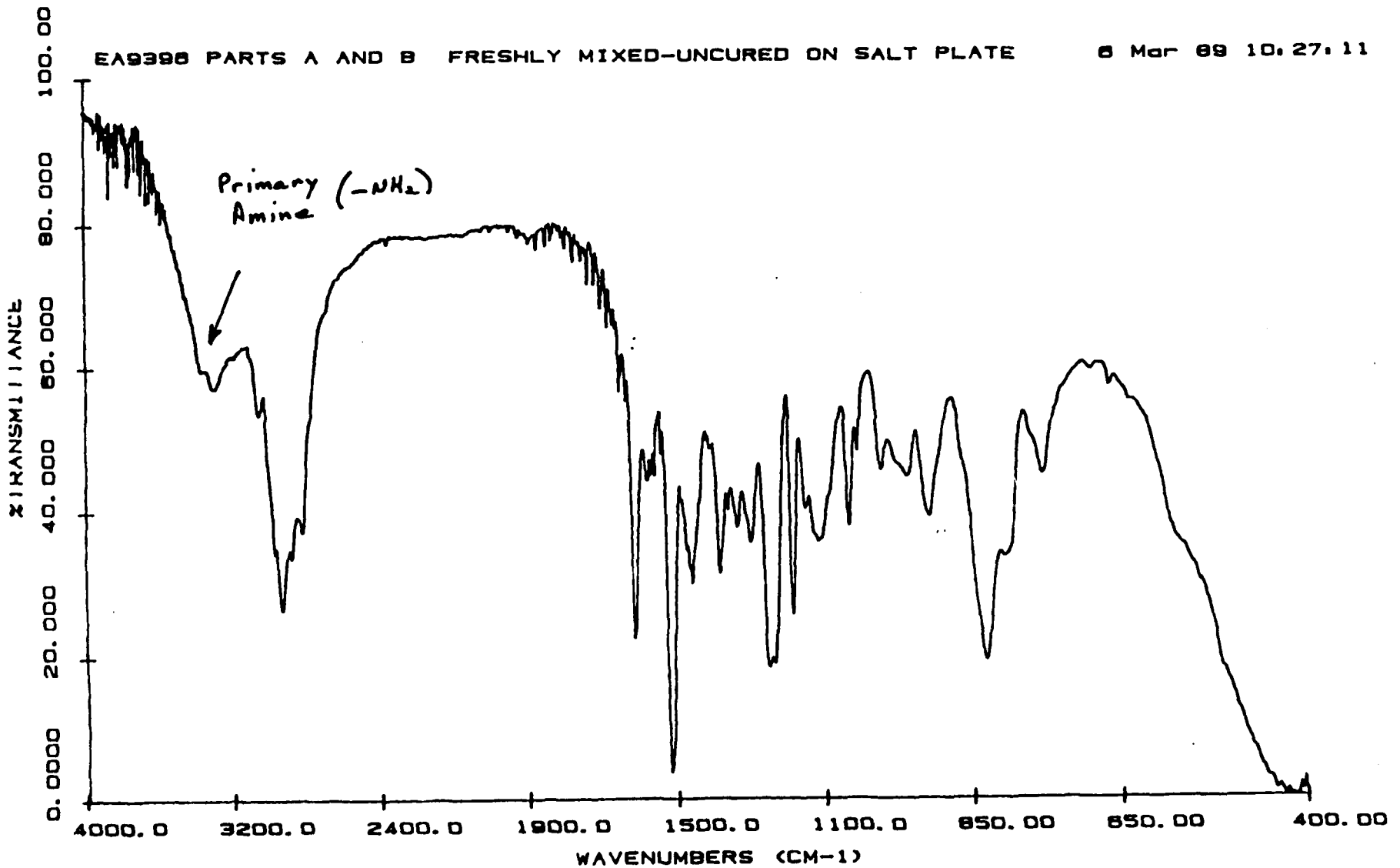


Figure 13. FTIR Spectra of Freshly Mixed EA9396.

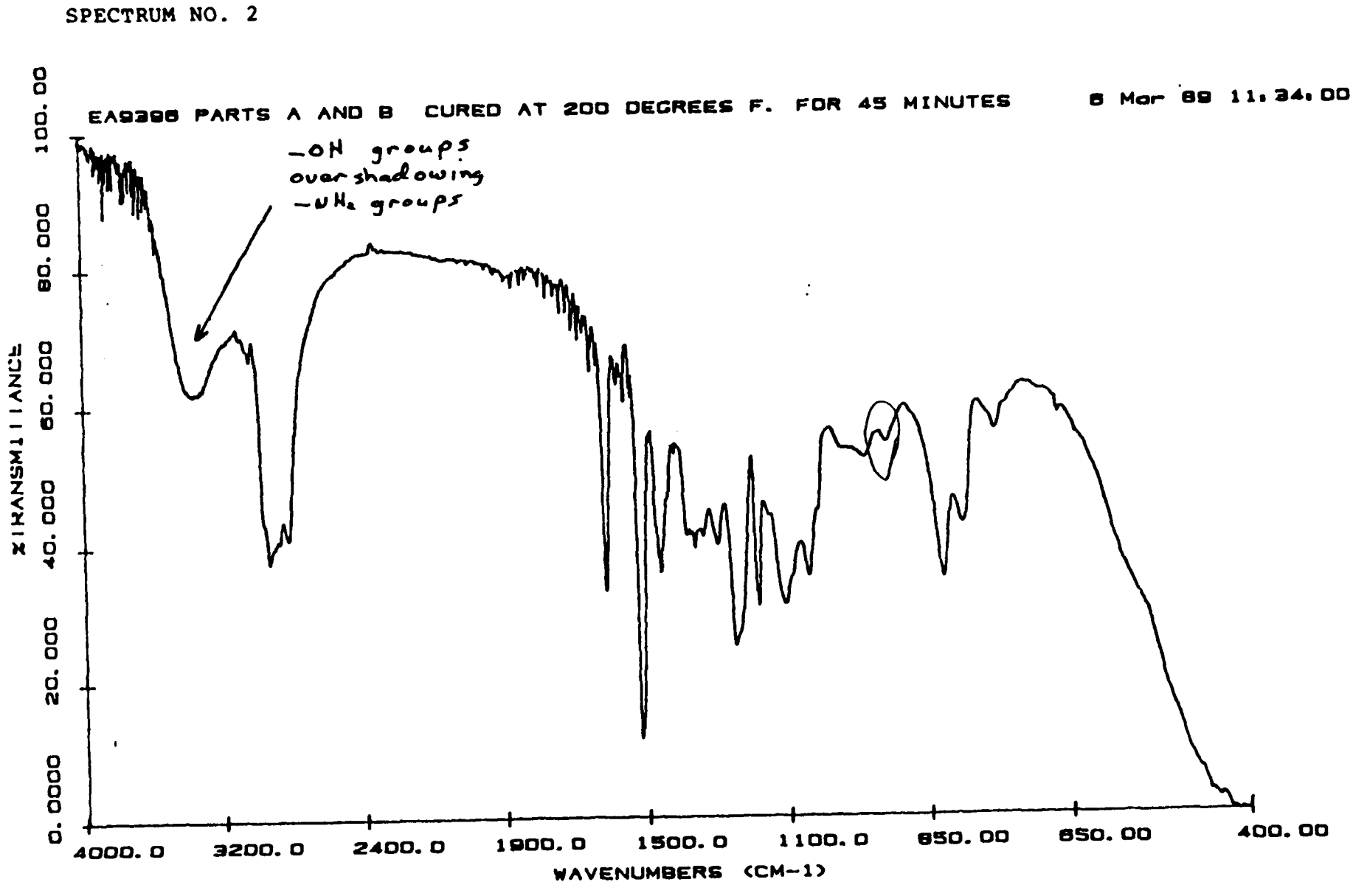


Figure 14. FTIR Spectra of EA9396 after 45-Minute, 93°C (200°F) Cure.

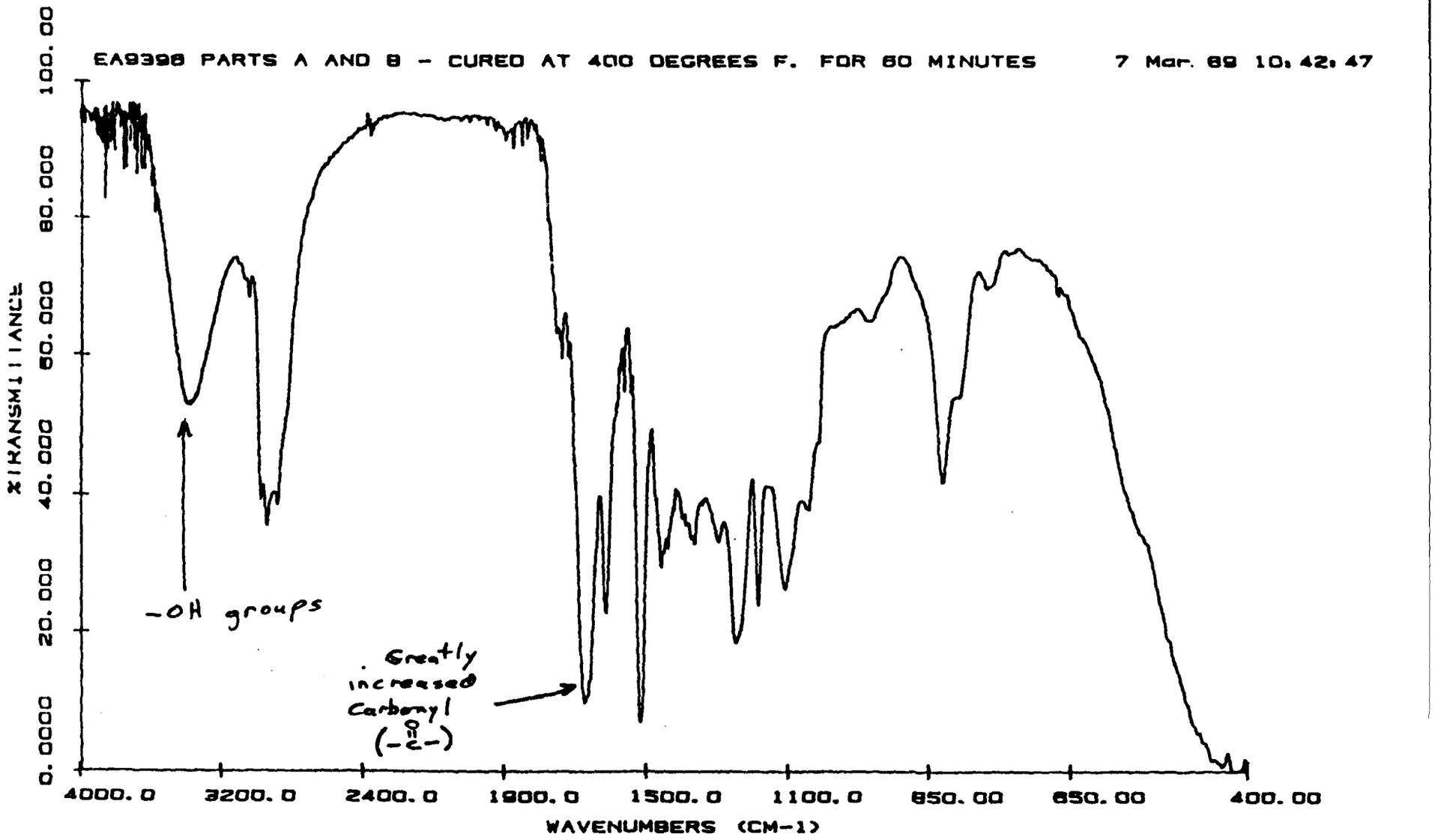


Figure 15. FTIR Spectra of EA9396 after 60-Minute , 204°C (400°F) Postcure.

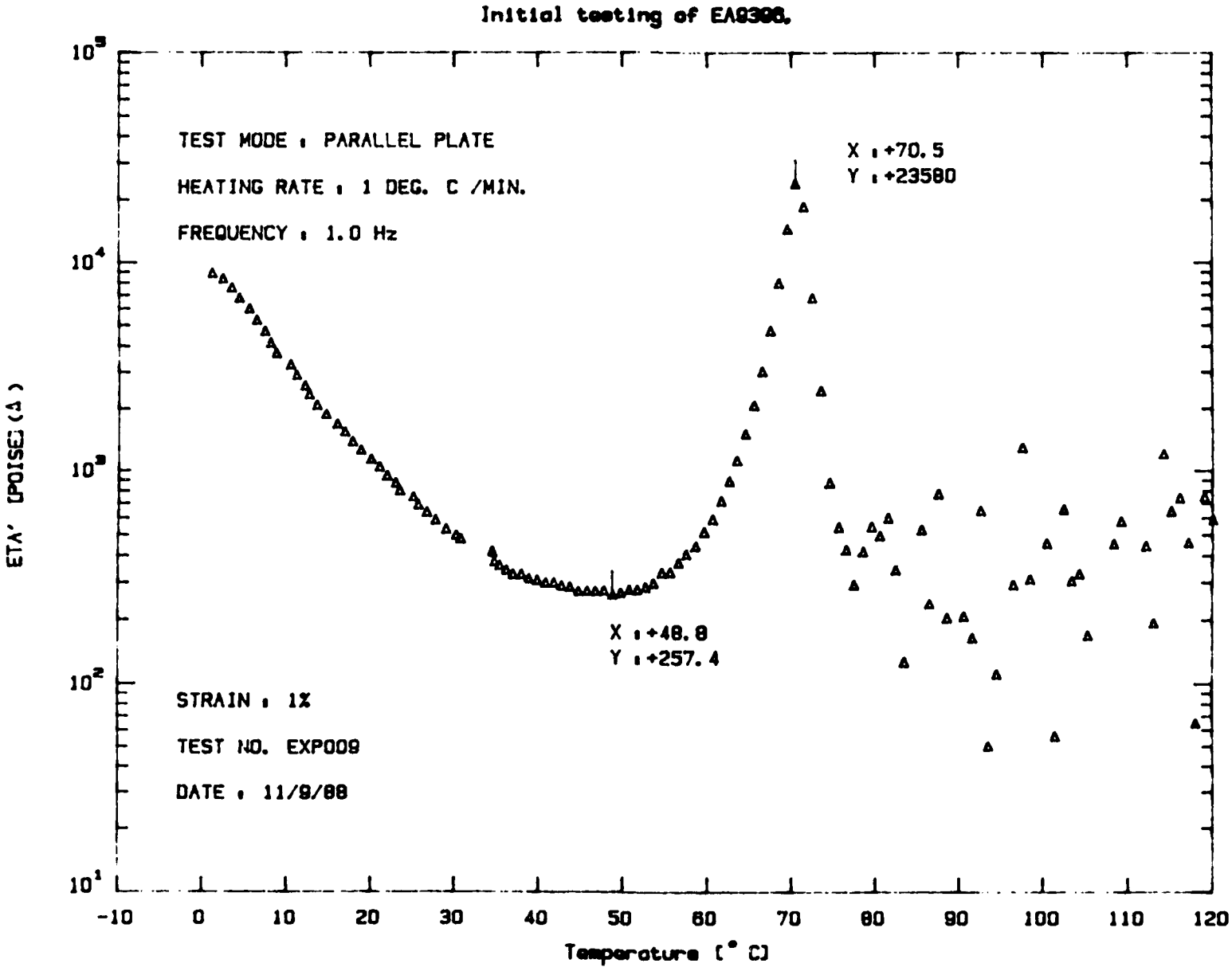


Figure 16. Viscosity Cure Profile for Fresh EA9396.

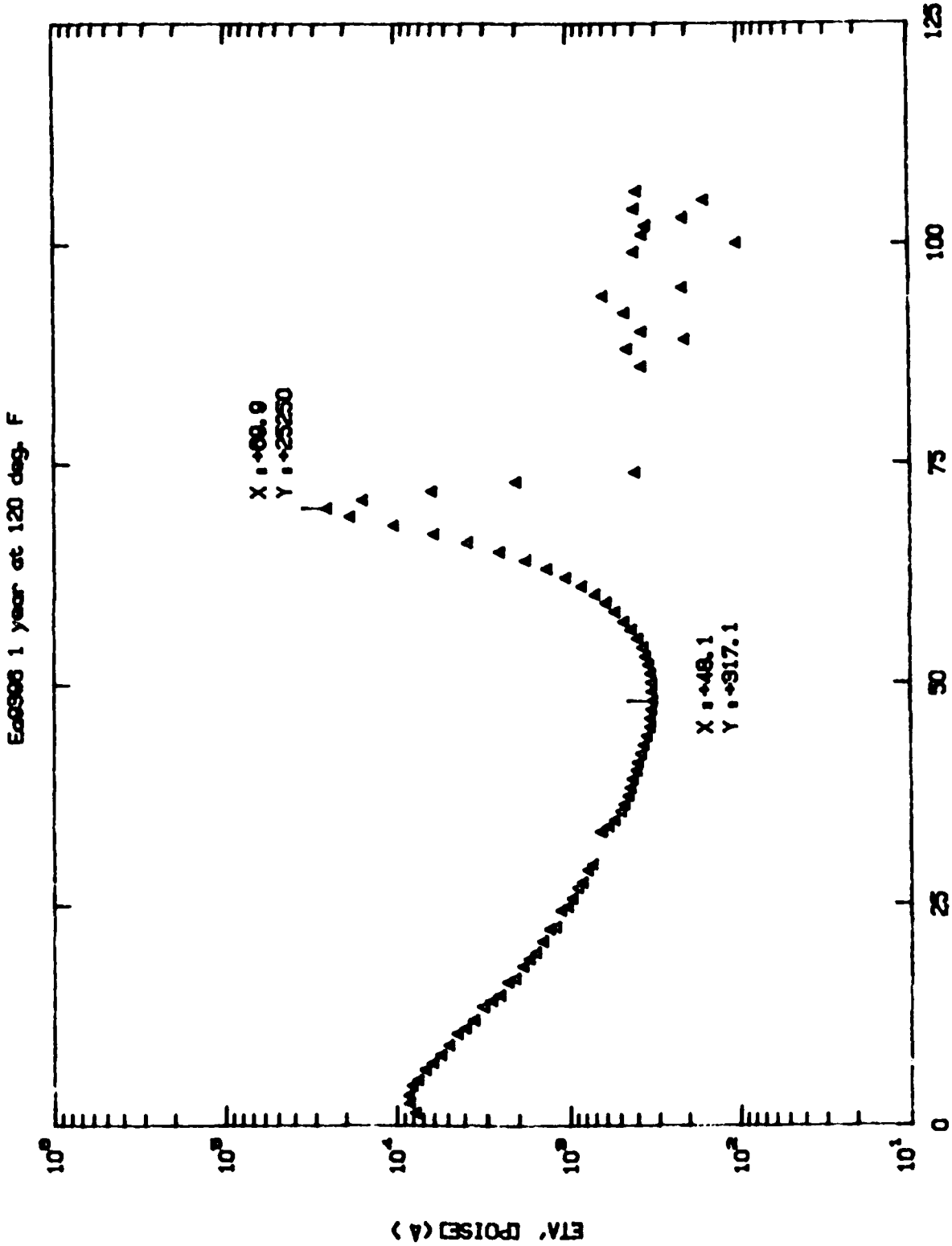


Figure 17. Viscosity Cure Profile for EA9396 Stored at 120°F for 12 Months.

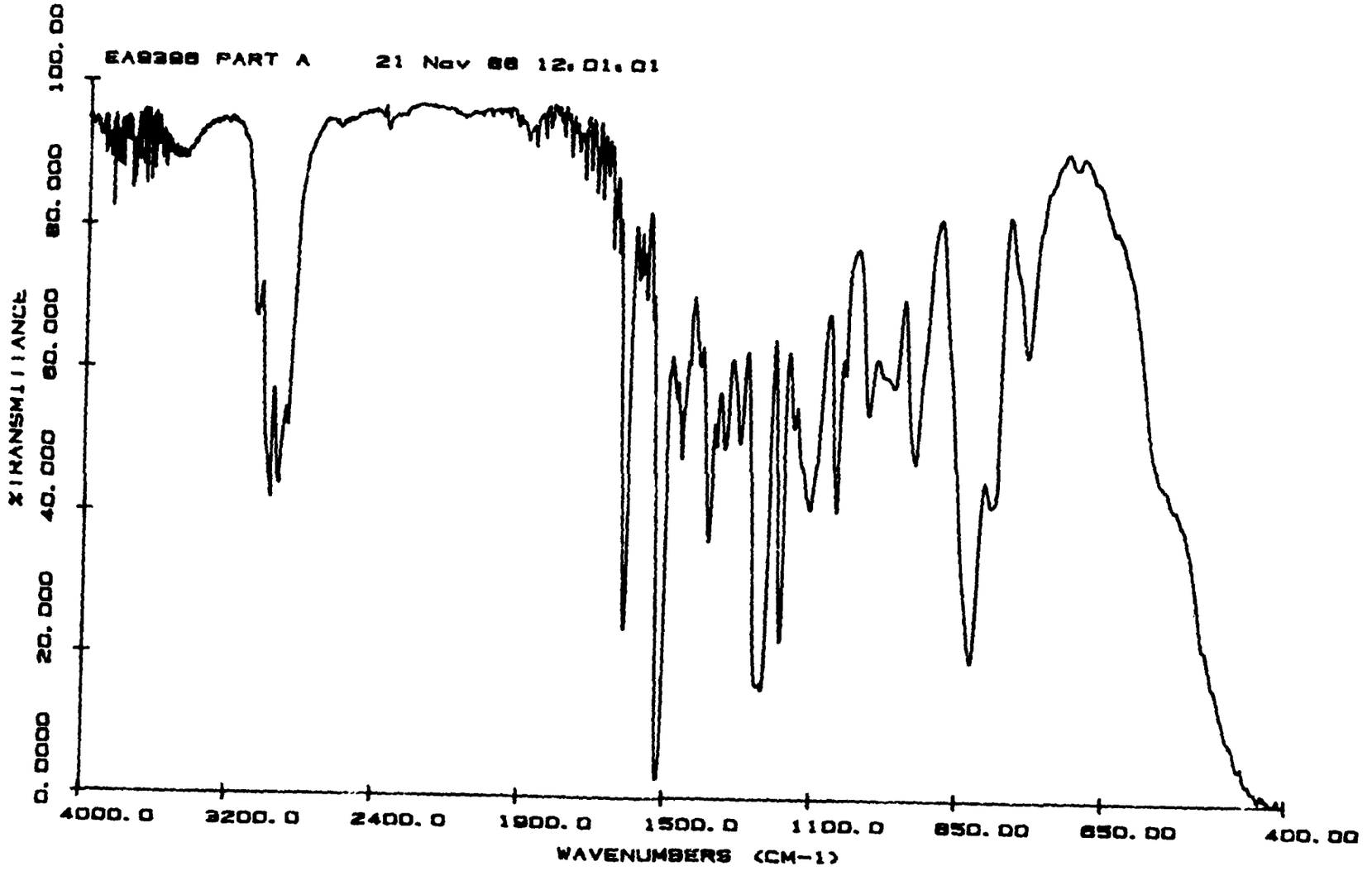


Figure 18. FTIR Spectra of Fresh EA9396/Part A Epoxy Resin.

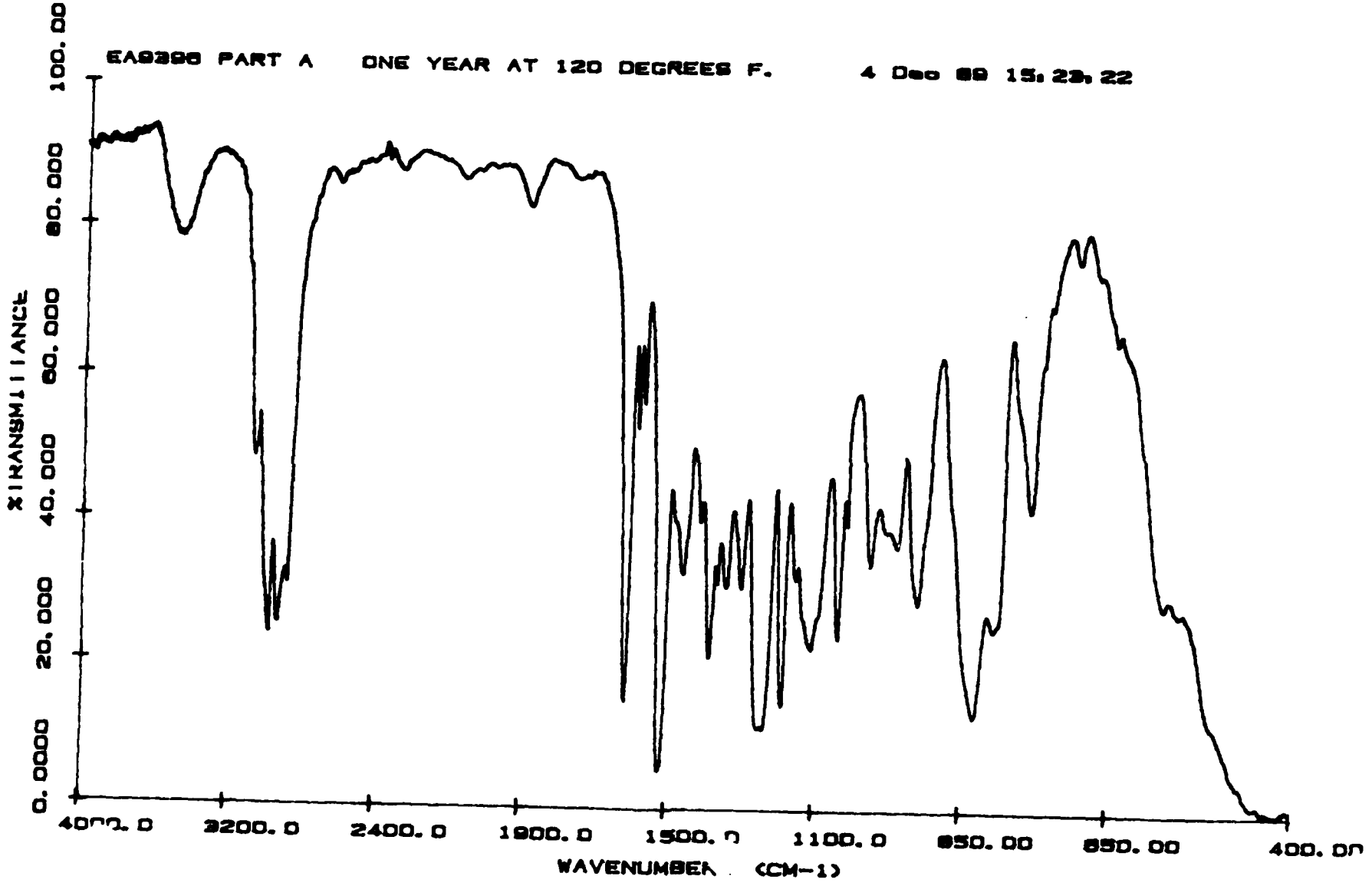


Figure 19. FTIR Spectra of EA9396/Part A Epoxy Resin After Storage at 120°F for 12 Months.

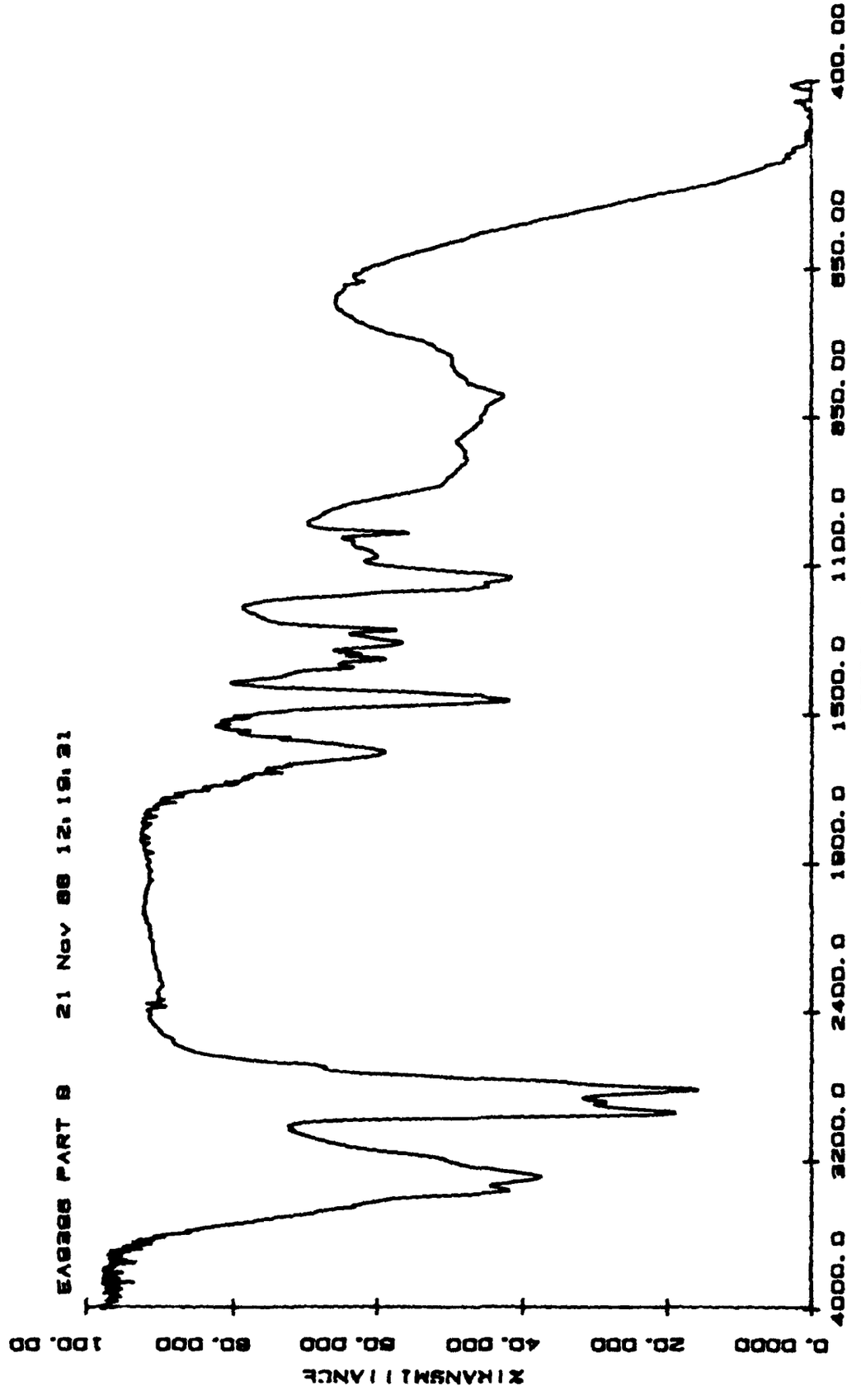


Figure 20. FTIR Spectra of Fresh EA9396/Part B Epoxy Resin.

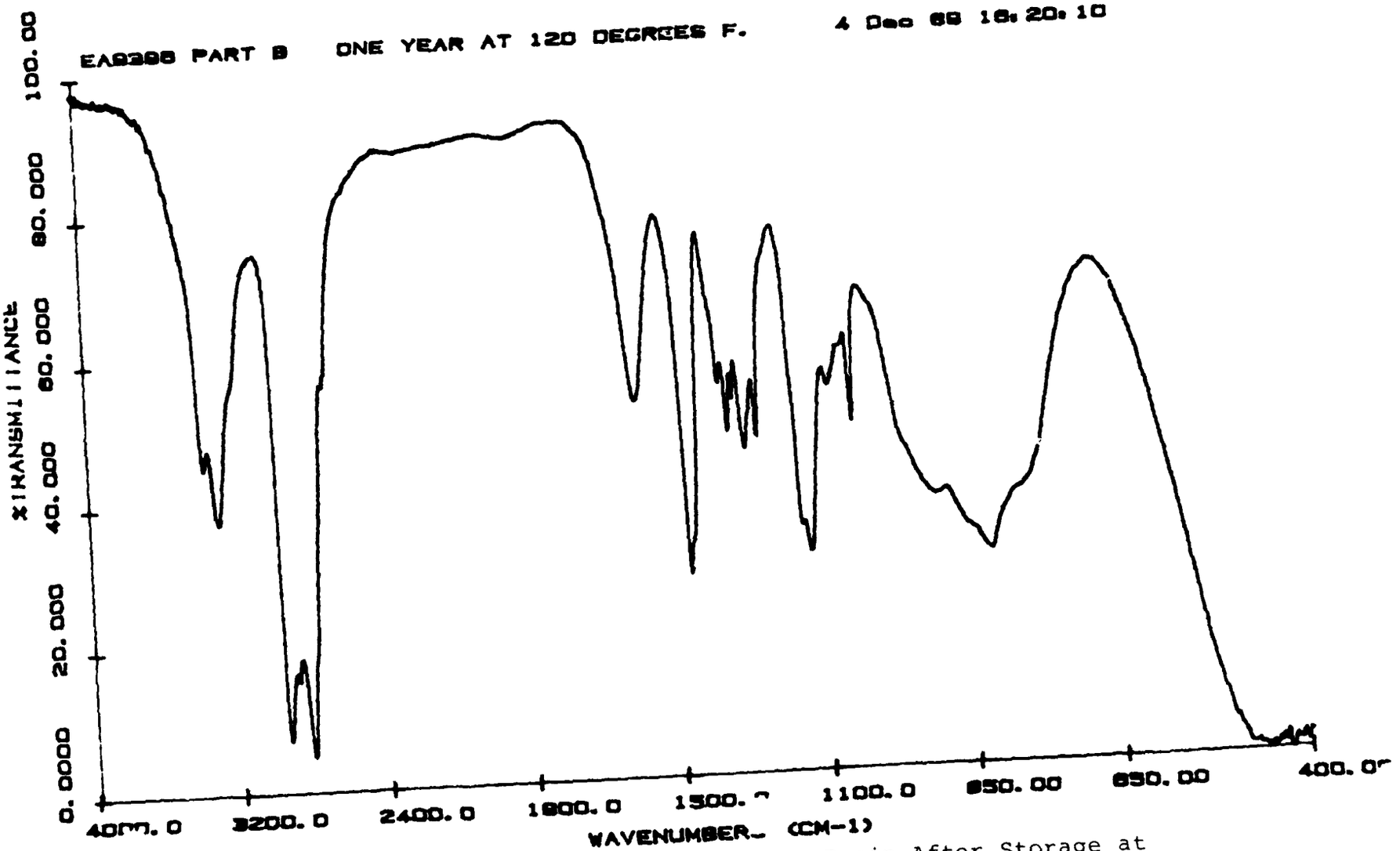
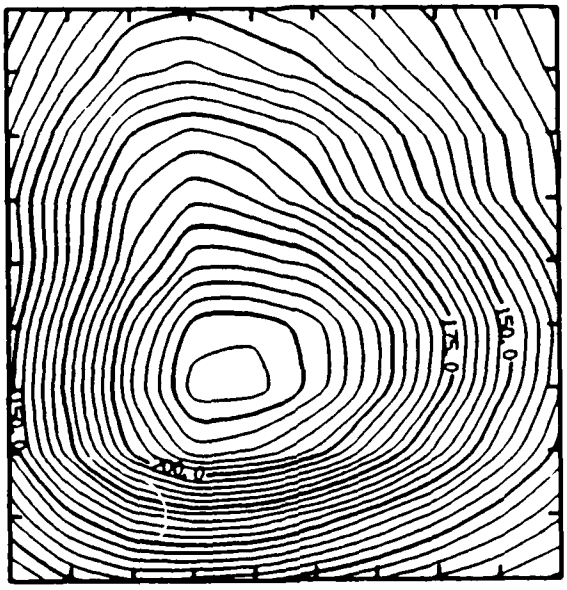
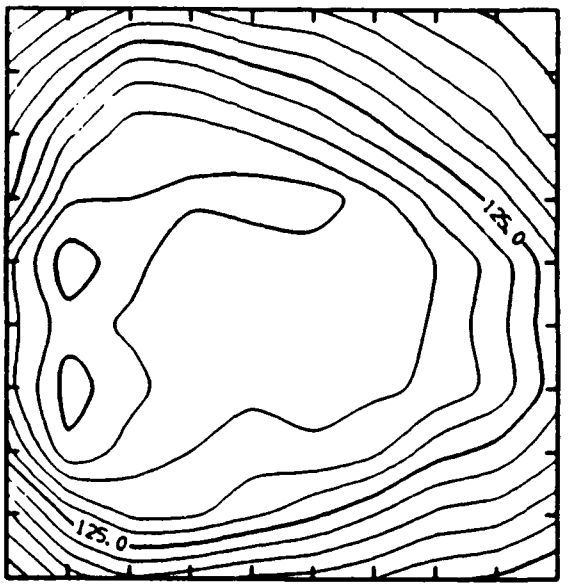


Figure 21. FTIR Spectra of EA9396/Part B Epoxy Resin After Storage at 120°F for 12 Months.



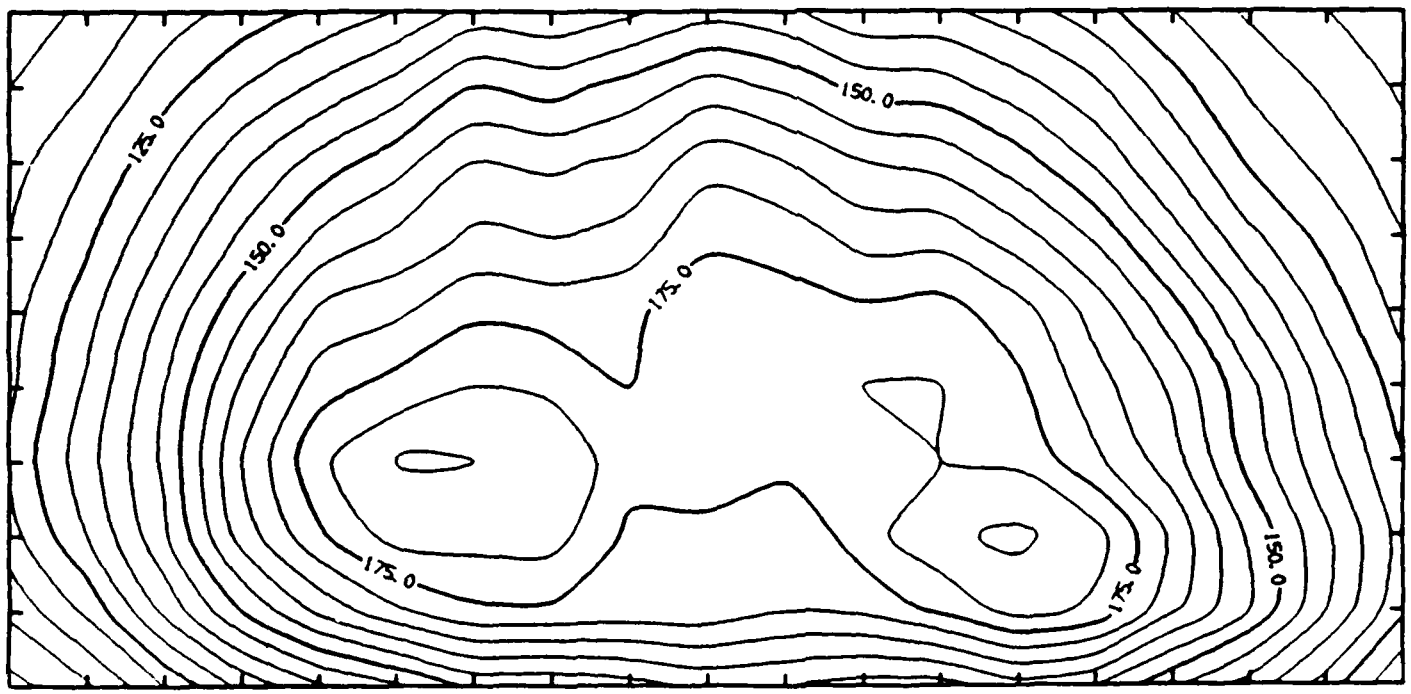
NOTE: Each contour represents a 5°F temperature increment and each hash mark represents one inch

Figure 22. Single White Lamp Test From 11 Inches.



NOTE: Each contour represents a 5°F temperature increment and each hash mark represents one inch

Figure 23. Single Red Lamp Test From 16 Inches.



NOTE: Each contour represents a 5°F temperature increment and each hash mark represents one inch

Figure 24. Twin Lamp Test From 16 Inches Spaced 9 Inches Apart.

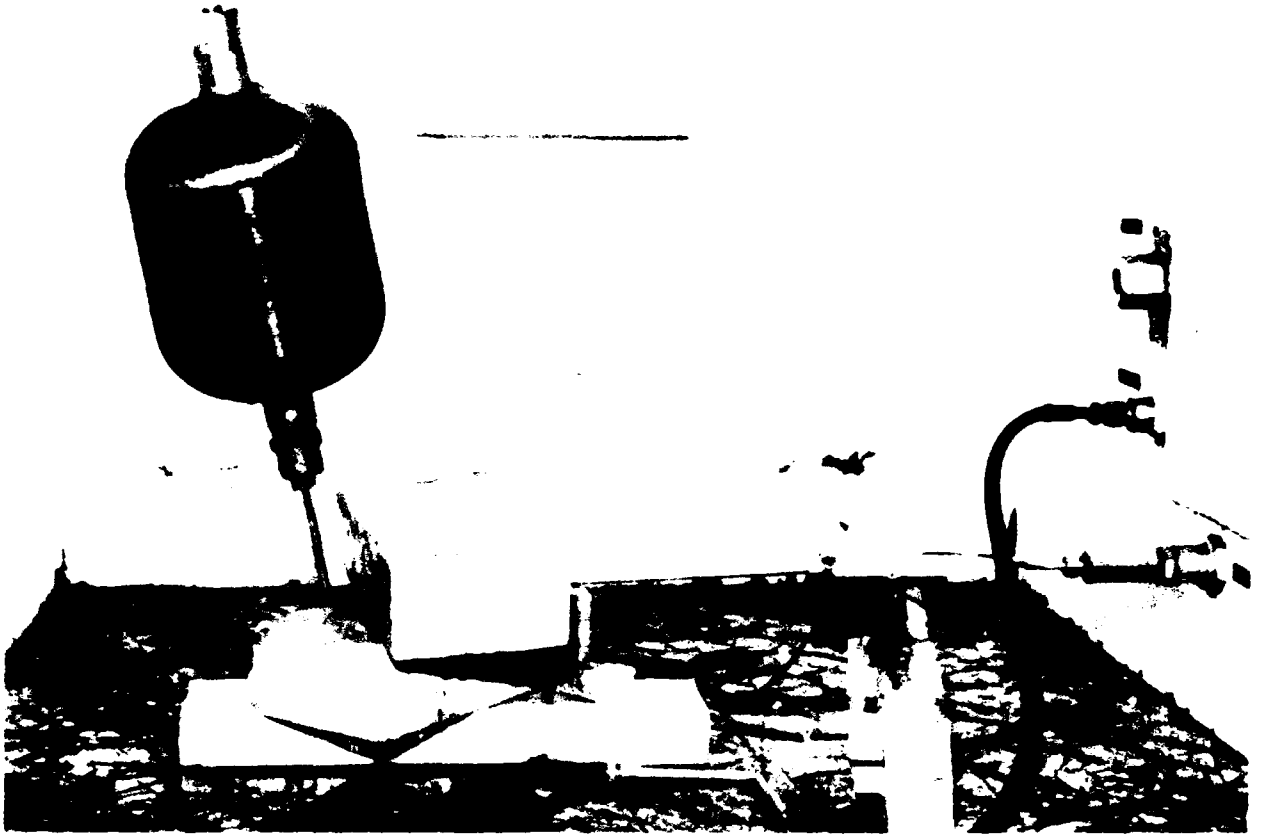


Figure 25. Typical Wet Wound Pressure Bottle Before Hydroburst.

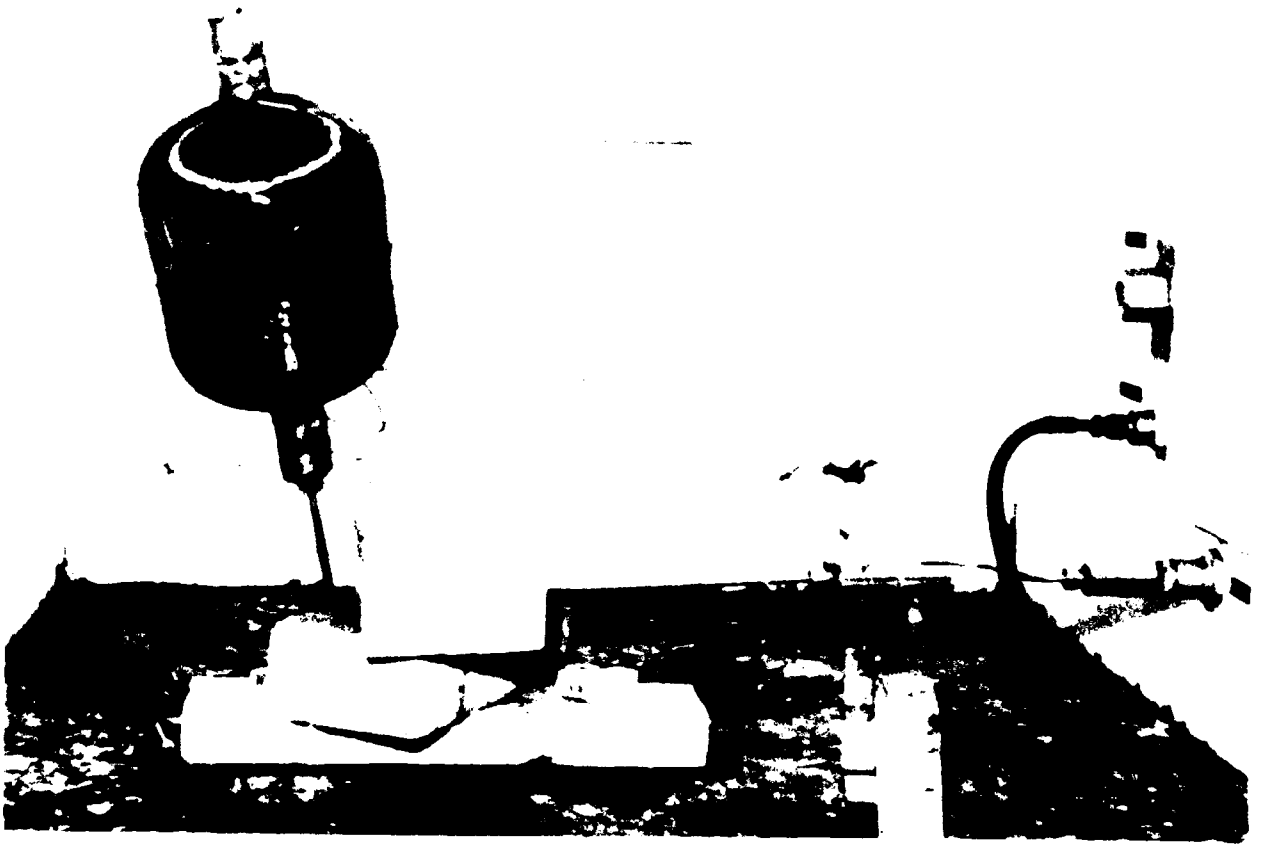


Figure 26. Typical Wet Wound Pressure Bottle After Hydroburst.

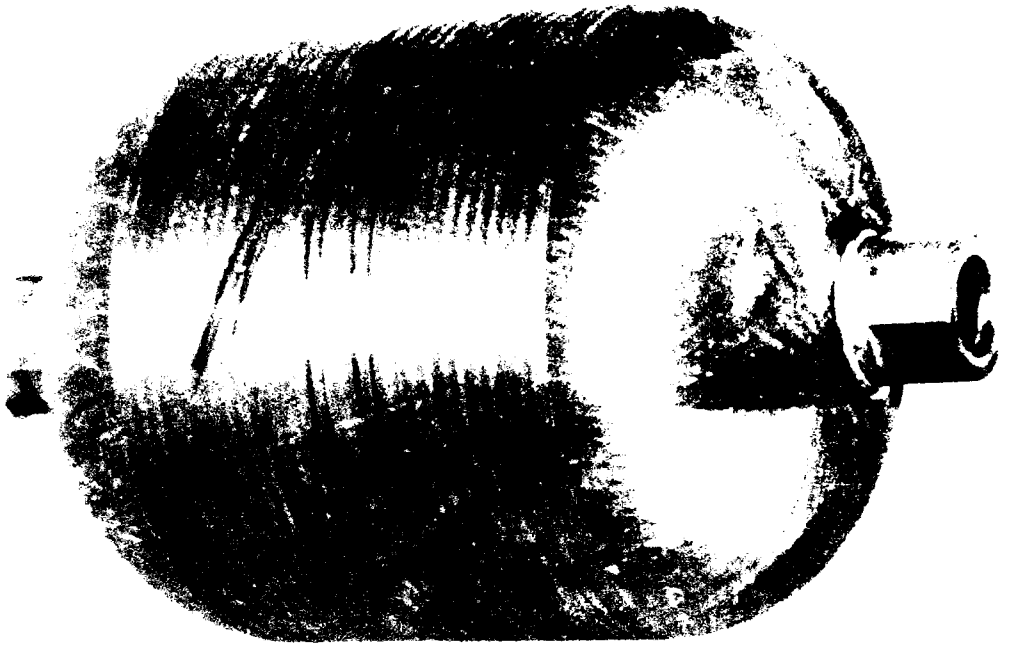


Figure 27. Resin Transfer Molded Pressure Vessel After Unsuccessful Burst Attempt.

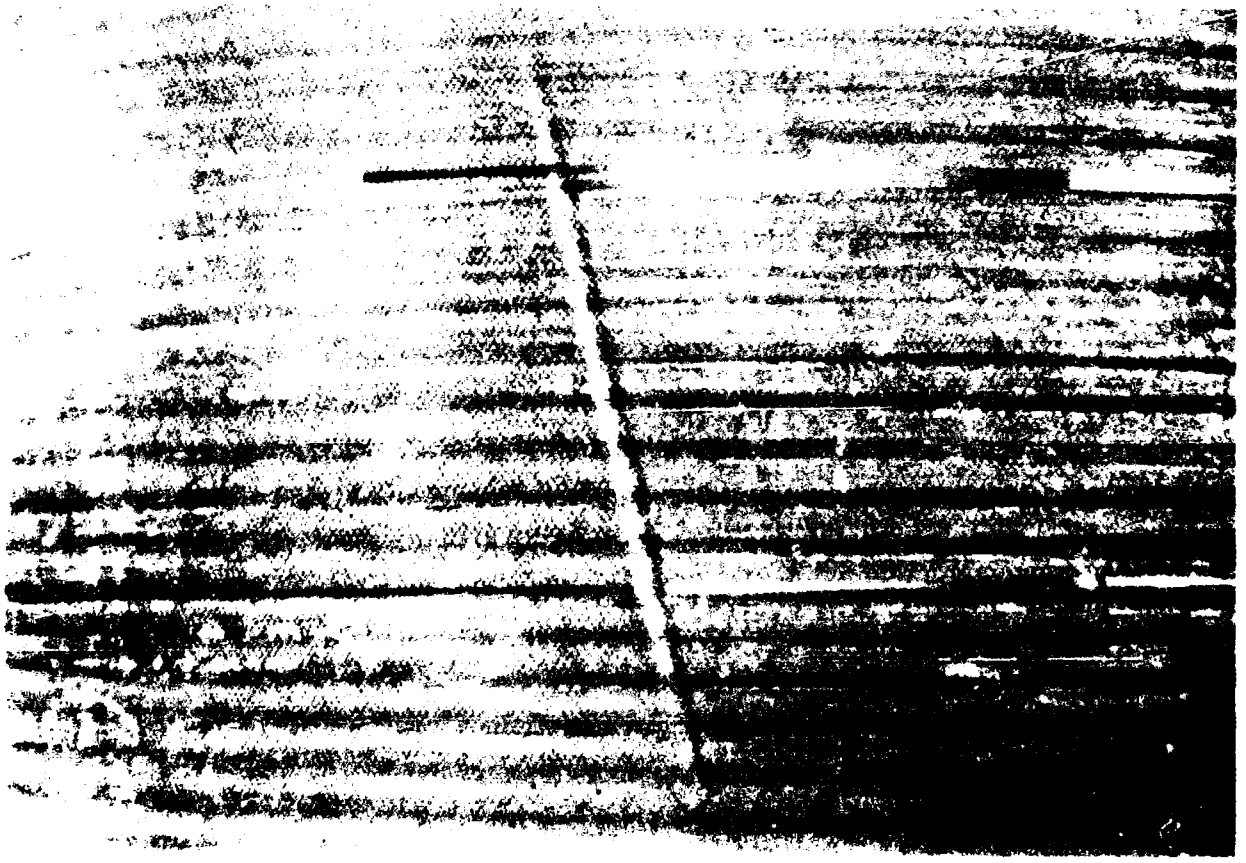


Figure 28. Close-Up of Failed Hoop Fibers Initiated by Wrinkle.

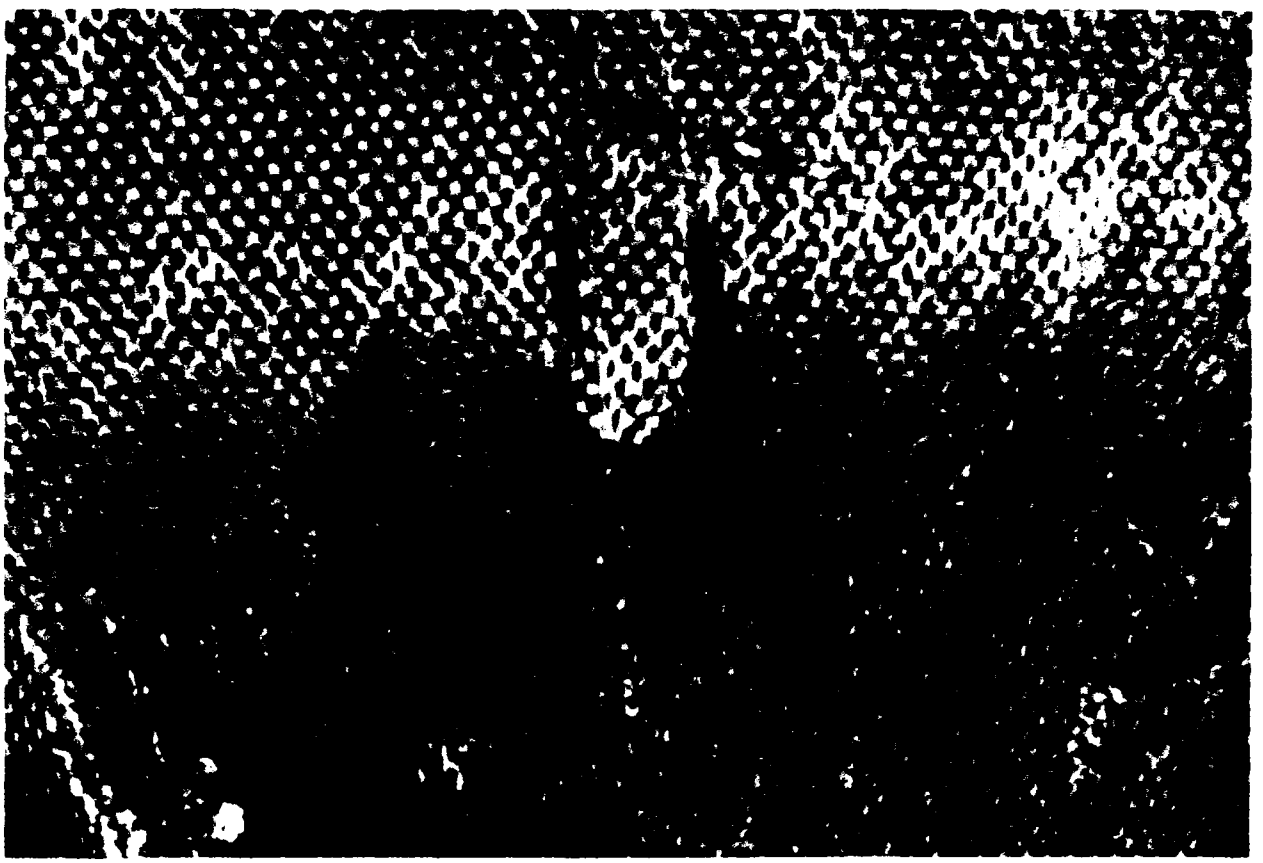


Figure 29. Fractured Tow in Dome Area of Resin Transfer Molded Pressure Vessel.

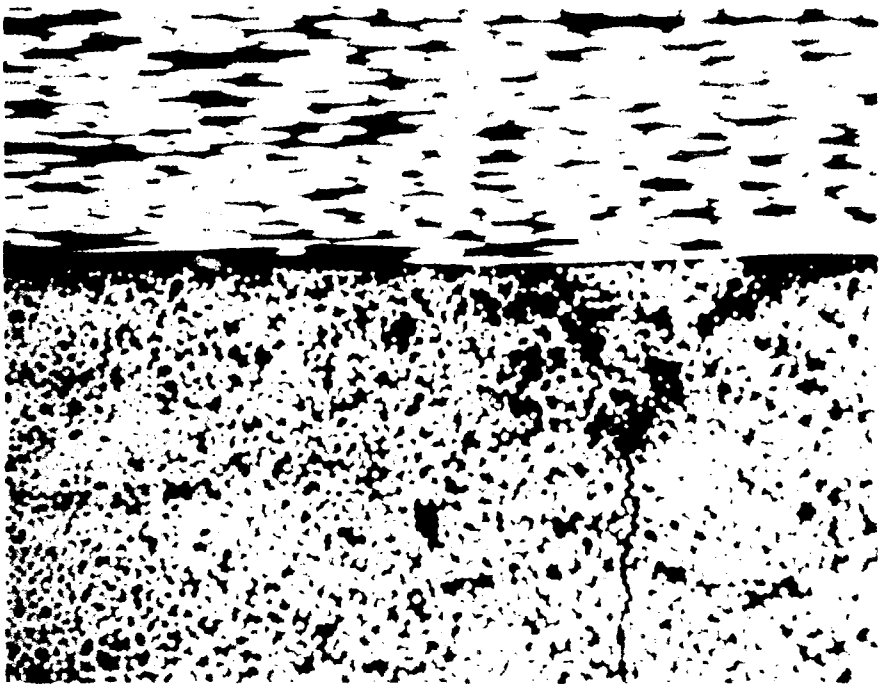


Figure 30. Hoop Area in Wet Wound Bottle.

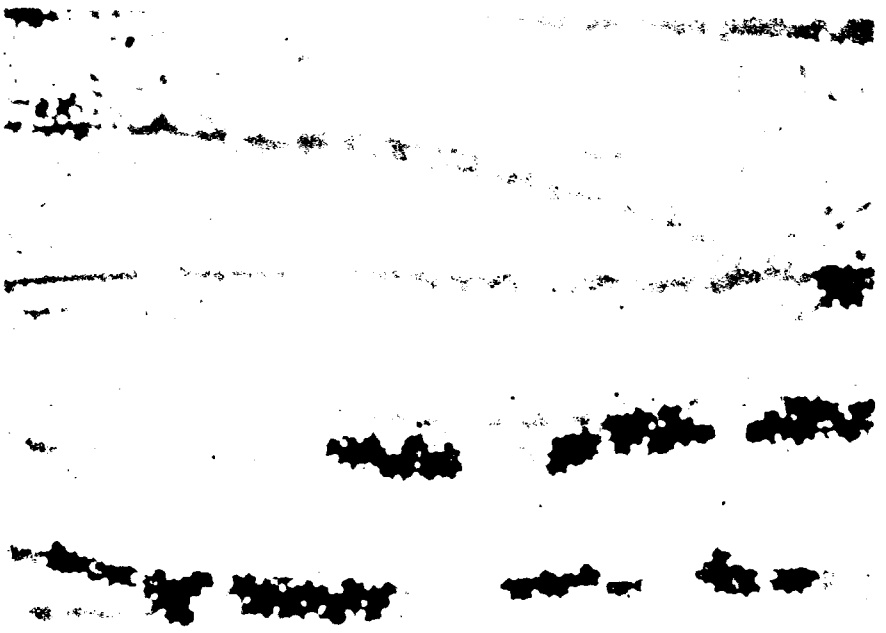


Figure 31. Dome Area in Wet Wound Bottle.

Figure 32. Hoop Area in Resin Transfer Molded Bottle.

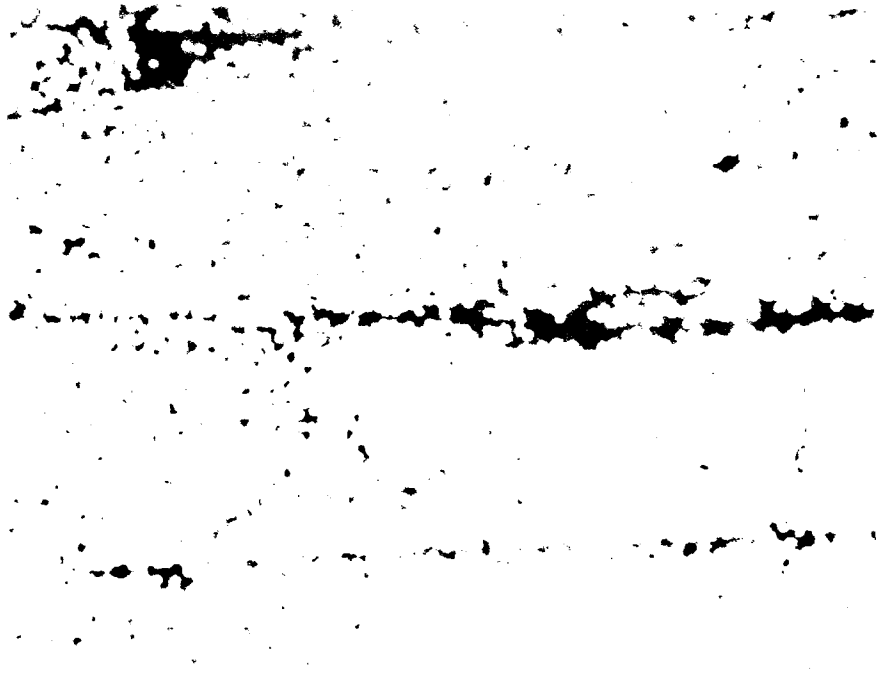


Figure 33. Dome Area in Resin Transfer Molded Bottle.

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